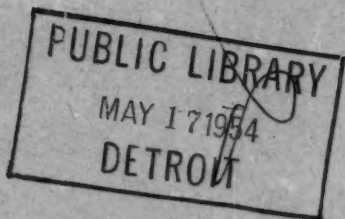


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# ANALYTICAL ABSTRACTS

Dealing with all branches  
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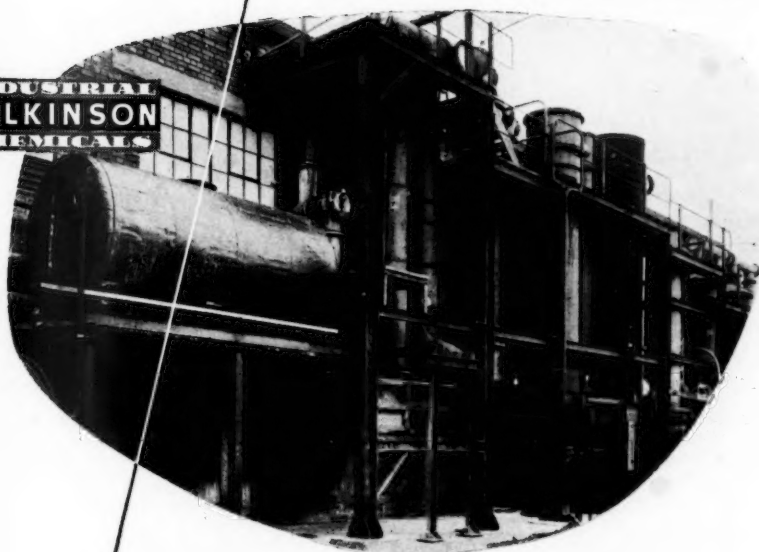
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## ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

632. **Rapid filtration and drying technique.** R. H. Pierson (*Anal. Chem.*, 1953, 25 [12], 1939).—Solutions containing partly hydrolysed cellulosic solids or other gelatinous ppt. can be filtered rapidly through a paper thimble supported in a filter tube. Moderate suction may be applied if the thimble is cushioned with a plug of glass wool. The ppt. can be dried by placing the tube on its side in an oven and drawing heated oven air through it by means of a rubber tube attached to the small end of the filter tube. J. M. JACOBS

633. **Determination of the purity of chemical substances by the phase rule.** H. A. Frediani (*Rendiconti Ist. Sup. Sanit.*, 1953, 16, 502-510).—The method, which is based on the solubility of a mixture in a solvent, is for determining the degree of purity of a compound and possibly for obtaining small quantities of one of two compounds in a state of purity. The method has been used successfully for a series of amino-acids and especially for use in the study of different optical isomers. A table giving the solubility of 20 different amino-acids at 30° C is provided. P. HAAS

634. **Titration curve for argentometric determination.** J. E. Ricci (*Anal. Chem.*, 1953, 25 [11], 1650-1656).—The shape of the curve of  $p[Ag^+]$  in the titration of KCN with  $AgNO_3$  was investigated to determine the position of the inflection point relative to the equivalence point and to the point of first appearance of a ppt. Two sets of conditions were considered, (i) titration of aq. KCN with  $AgNO_3$ , in which the pH is variable during the titration, and (ii) titration of KCN in presence of excess of aq.  $NH_3$ , during which the pH value is almost constant. Equations are derived for the calculation of the equilibrium constants from the characteristics of the titration curve. G. P. COOK

635. **Universal anticipation of end-point system for automatic titrations.** W. N. Carson, jun. (*Anal. Chem.*, 1953, 25 [11], 1733-1735).—A means of preventing over-titration in automatic titrations is described. The over-titration, caused by time lag in the response of the indicator system, is overcome by withdrawal of a portion of the sample, titration of the remainder at a fast rate to the end-point, return of the portion and titration slowly to the end-point. Variation in the amount of sample withdrawn and in the slow titration rate permits specific adaptation. The system, as designed for an automatic titrator, when applied to the titration of dichromate samples shows a standard deviation of < 1.0 per cent. G. P. COOK

636. **Reverse titrations for economy of materials and time and improvement of accuracy in volumetric analysis.** F. L. Hahn (*Anal. Chim. Acta*, 1953, 9 [5], 400-407).—A titration system is described in which both solutions are measured from burettes. After

reaching the first end-point a small excess of the titrant is added; this excess is titrated to a second end-point with the first solution, and an excess of this soln. is added for a further titration. An average is calculated from the successive titres and the reverse titrations are repeated until a sufficient number of results are obtained to give an average titre of the desired accuracy. W. C. JOHNSON

637. **Studies on the velocity of flow of solvents of some homologous series through chromatographic columns in relation to their viscosity, height of the column and density.** D. R. Gupta and A. K. Bhattacharya (*J. Indian Chem. Soc.*, 1953, 30 [9], 661-664).—The experimental investigation reported shows that the velocity of flow of a liquid through an adsorbent column is related to the height of the column and the density of viscosity of the liquid, that the expression  $Vh\eta/td$  ( $V$  = vol.,  $h$  = height of column,  $\eta$  = viscosity,  $t$  = time of flow and  $d$  = density) is constant for the members of homologous series, and that the constant differs in value for different groups of compounds, such as alcohols, ketones, esters or aromatic hydrocarbons. O. M. WHITTON

638. **Determination of traces of an element in a powder without the use of standard samples.** J. Gillis and J. Eeckhout (*Spectrochim. Acta*, 1953, 5 [6], 409-416).—A new spectrochemical method is described for the determination of an unknown element in a sample of powder without the use of standard samples. From the spectra obtained at several dilutions of the sample, the slope of the standard curve can be determined. The determination of the relative intensities of the spectral lines obtained after the addition of known amounts of the element to the sample allows the content of the element in the sample to be determined by extrapolation. The determination of Mg in graphite from nodular cast iron is given as an example, and a curve has been constructed for the range 0.1 to 1 per cent. of Mg. N. E.

639. **Calculation of amount of tracer carried with precipitates of its radioactive parent.** F. W. Lima (*Anal. Chem.*, 1953, 25 [12], 1924-1925).—The method of calculating the proportion of measured radioactivity due to each nuclide when genetically related radio-isotopes are used to follow a chemical reaction (*cf.* Kirby, *Anal. Chem.*, 1952, 24, 1678) is extended to the calculation of the carrying of such isotopes when they are in equilibrium before fractionation by pptn. J. M. JACOBS

640. **Two-dimensional paper chromatography of radioactive substances.** F. P. W. Winteringham (*Nature*, 1953, 172, 727-728).—An automatic scanning technique and a Geiger-Müller tube for the location and estimation of radioactive substances prepared on unidimensional paper chromatograms has been described earlier (*Brit. Abstr. C*, 1952, 282). The application of this technique to two-dimensional

chromatograms is tedious because a large sheet of paper has first to be cut up into the equivalent of unidimensional strips and each strip has to be scanned separately. To obviate this difficulty, an alternative technique to the conventional form of two-dimensional paper chromatography has been successfully devised for the application of two or more solvent systems to the same labelled mixture. The method is described in detail. B. VINEY

641. Tabulated diffraction data for hexagonal isomorphs. L. K. Frevel and H. W. Rinn (*Anal. Chem.*, 1953, 25 [11], 1697-1717).—One hundred and seventeen hexagonal prototype structures are tabulated, and 1287 hexagonal substances are arranged according to the ascending order of their axial ratios. Powder diffraction patterns for 53 hexagonal structures are also included.

G. P. COOK

642. Tetraphenylphosphonium and tetraphenylstibonium chlorides as analytical reagents. H. H. Willard and L. R. Perkins (*Anal. Chem.*, 1953, 25 [11], 1634-1637).—Tetraphenylphosphonium chloride in the presence of NaCl quant. precipitates complex chloride salts of  $\text{Sn}^{IV}$ , Cd or Hg, such as  $[(\text{C}_6\text{H}_5)_4\text{P}]_2\text{SnCl}_6$ . These salts cannot be weighed because NaCl must be used in the wash soln. Excess of reagent can be titrated potentiometrically with standard iodine soln. to form  $(\text{C}_6\text{H}_5)_4\text{P}^+\text{I}_4^-$ . Tetraphenylphosphonium permanganate, perphenate and perchlorate are insoluble in cold water and therefore both gravimetric and titrimetric methods are possible. For periodate, the titrimetric method only can be used. Tetraphenylstibonium chloride may be used for the gravimetric determination of permanganate and perchlorate, but with the latter the low solubility of the reagent makes the method impractical. Some details of the methods are given. The salts are soluble in  $\text{CHCl}_3$ . The average error under the recommended conditions is about  $\pm 0.06$  mg. It can be predicted that tetraphenylphosphonium chloride may also be used to precipitate Pt, Te, Tl and technetium. The alkali metals, alkaline earths, Al,  $\text{Mn}^{II}$ ,  $\text{Cr}^{III}$ , Ni, Co, Zr, sulphate, borate, phosphate, carbonate, acetate, tartrate and citrate are not precipitated and do not interfere. The following ions are precipitated either completely or partially: permanganate, perphenate, periodate, perchlorate, persulphate, fluoroborate, chromate, molybdate, tungstate, thiocyanate, iodide,  $\text{Sn}^{IV}$ , Cd, Zn, Pt,  $\text{Fe}^{III}$ , Au, Sb, Bi, Ti,  $\text{Ti}^{III}$ ,  $\text{Te}^{IV}$  and  $\text{UO}_2^{++}$ . The interference from Sn was obviated by complex formation with acid tartrate, from  $\text{Fe}^{III}$  by phosphate and phosphoric acid, from Ti by metaphosphoric acid and, in the iodine titration, from Cu by citrate.

O. M. WHITTON

## 2.—INORGANIC ANALYSIS

643. Separation of lithium, sodium and potassium present as sulphates and citrates by means of paper chromatography. D. P. Burma (*Anal. Chim. Acta*, 1953, 9 [6], 513-517).—The unidimensional descending technique is used and the apparatus is similar to that of Burstall *et al.* (*Brit. Abstr. C*, 1951, 36). Mixtures of water with methanol, ethanol or acetone are used for development. Sulphate spots are made visible by spraying with a 0.2 per cent. soln. of  $\text{BaCl}_2$  in 70 per cent. ethanol, drying and spraying with a 0.05 per cent. soln. of

Na rhodizonate in 50 per cent. ethanol; the spots then appear yellow on a reddish background. Citrate spots are sprayed with a soln. of 50 mg of bromophenol blue in 100 ml of 70 per cent. ethanol; the spots then appear blue on a yellow background.  $R_f$  values are determined under various conditions of development. The proportion of water in each developing solvent influences the separation of mixtures of the salts, and the experiments indicate the optimum conditions. W. C. JOHNSON

644. The determination of lithium in magnesium-lithium alloys by internal standard flame photometry. (The late) A. M. Robinson and T. C. J. Ovenston (*Analyst*, 1954, 79, 47-50).—The use of the double-beam flame photometer previously described (*Brit. Abstr. C*, 1952, 120) for determination of 11 to 14 per cent. of Li in Mg-Li alloys is described. The accuracy is  $\pm 1$  per cent. The internal standard technique is used, K being the standard selected. Na and small amounts of K in the alloys do not interfere and Ag, Zn, Al and Cd if totalling  $> 10$  per cent. of the alloy have no effect. A. O. JONES

645. Determination of potassium with a lithium cobaltinitrite reagent. T. Dupuis (*Anal. Chim. Acta*, 1953, 9 [6], 493-499).—Pptn. of K with Na cobaltinitrite yields K Na salts of various compositions. Li cobaltinitrite yields the pure K salt. The reagent is prepared by mixing a solution of 2.7 g of lithium nitrite in 9 ml of water with a soln. of 1.5 g of cobalt nitrate made up to 14 ml with water and acidified with 0.6 ml of acetic acid; the excess of nitrous gases is removed by drawing air through the soln. The reagent must be prepared freshly each day. Twenty ml are required for 0.1 g of KCl and the pptn. can be carried out between 25° and 55° C. The ppt. is washed with 10 per cent. acetic acid, ethanol and ether, dried at 110° C and weighed as (hygroscopic)  $\text{K}_2\text{Co}(\text{NO}_2)_2 \cdot 2\text{H}_2\text{O}$  or, when K  $> 20$  mg, heated to 400° C and weighed as a mixture of  $\text{CoO} + 3\text{KNO}_3$ . The mean error is  $\pm 0.7$  per cent. Mg,  $\text{NH}_4^+$ , Rb and Cs salts are precipitated by the reagent and Na is co-precipitated with K. Alternatively the  $\text{NO}_2^-$  in the ppt. can be titrated by dissolving it, after washing with 10 per cent. acetic acid and removing the acid with a little water, in 0.02 N  $\text{KMnO}_4$  containing 20 per cent.  $\text{H}_2\text{SO}_4$  heated to  $> 60^\circ\text{C}$ , adding an excess of 0.02 N Na oxalate and back-titrating with  $\text{KMnO}_4$ . On a micro scale (0.5 to 1.0 mg of K) the error is  $\pm 1.0$  per cent. In a spectrophotometric method, nitroso-R salt was used to determine the Co in the ppt. either after the  $\text{NO}_2^-$  had been destroyed with  $\text{H}_2\text{O}_2$  or by boiling with HCl.

W. C. JOHNSON

646. Spectrophotometric determination of potassium with dipicrylamine. E. Gastinger (*Z. anal. Chem.*, 1953, 140 [5], 335-342).—In contrast to the Li, Na, Mg and Ca salts of dipicrylamine, the K salt is almost insoluble in water at 0° C. After separation, the salt is dissolved in acetone and the extinction of the soln. buffered with  $\text{NaHCO}_3$  and  $\text{Na}_2\text{CO}_3$  is determined in a Pulfrich photometer having a mercury filter ( $\lambda = 436 \text{ m}\mu$ ). The concn. of acetone and buffer have no influence upon the extinction. The soln. is stable for at least 2 hr. and obeys the Beer-Lambert law in the range of concn. studied (up to 400  $\mu\text{g}$  of K per litre). The method detects concn. of K as low as  $4 \times 10^{-4}$  g per litre with an accuracy of  $\pm 2$  per cent.

S. K. LACHOWICZ

647. Volumetric determination of potassium and ammonium in the presence of each other. W. Rüdorff and H. Zannier (*Z. anal. Chem.*, 1953, 140 [4], 241-244).—Before the pptn. of K as  $\text{KB}(\text{C}_6\text{H}_5)_4$ , the  $\text{NH}_4^+$  are removed by conversion to hexamine with formaldehyde and estimated by titration of the acid produced. The soln. of the two cations should contain 10 to 20 mg of K and be neutral to methyl red. Ten ml of 30 per cent. aq. formaldehyde, neutralised to phenolphthalein with 0.05 N NaOH, are added, and the  $\text{NH}_4^+$  is estimated by titration with carbonate-free 0.05 N NaOH. More aq. formaldehyde is added (in all 2 to 2.5 ml for each 1 mg  $\text{NH}_4^+$ ), and the alkalinity is made to 1 N with solid NaOH. The soln. is brought to the boil, and 0.2 per cent. "Kalignost" soln. is added with constant stirring. After cooling, the ppt. is filtered and washed first with  $\text{H}_2\text{O}$  saturated with  $\text{KB}(\text{C}_6\text{H}_5)_4$  and then quickly with distilled  $\text{H}_2\text{O}$ . The ppt. and filter are returned to the beaker, and acetone is added (not more than 20 to 25 ml for each 20 mg of K). After the addition of 1 to 2 ml of 2 N acetic acid, 1 ml of 0.1 N KBr and 2 drops of eosin soln., the soln. is titrated with 0.05 N  $\text{AgNO}_3$  soln. by the authors' previously described method (*Brit. Abstr. C*, 1953, 50).

J. H. WATON

648. Spectrophotometric determination of copper in ores with 2:2'-bipyridine (2:2'-dipyridyl). J. P. Mehlig and P. L. Koehrmstedt (*Anal. Chem.*, 1953, 25 [12], 1920-1921).—A cupric-ammonia complex is formed by addition of 6 M aq.  $\text{NH}_3$  to a Cu soln.; then 10 ml of 0.1 per cent. 2:2'-dipyridyl are added, followed by 1 ml of 10 per cent. hydroxylamine hydrochloride (to reduce the  $\text{Cu}^{2+}$  to  $\text{Cu}^+$ ) and 20 ml of "Methyl Carbitol" (diethylene glycol monomethyl ether) as a stabiliser. The soln. is diluted with water to 50 ml and the transmittance is measured at a wavelength of 430 m $\mu$ . The reagents must be added in the order given above and excess of  $\text{NH}_3$  must be avoided. In comparative tests with the iodide method in the determination of Cu in six ores, four oxides and two mattes, the difference between the two methods did not exceed  $\pm 0.09$  per cent., with an average difference of  $\pm 0.03$  per cent. Results were duplicated with a precision of  $\pm 0.01$  to  $\pm 0.11$  per cent. The results were also in good agreement with those obtained by the  $\text{NH}_3$ , triethanolamine and 1:10—phenanthroline spectrophotometric methods; the method can be applied to ores containing at least 22 per cent. of Cu. Interference occurs with  $\text{Cd}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$ , apparently by complex formation with the reagent.  $\text{CN}^-$  and  $\text{S}_2\text{O}_3^{2-}$  are the only common anions that interfere.

J. M. JACOBS

649. Applications of ion exchangers in analytical chemistry. XXVII. Separation of metals with anion exchangers in citrate form. O. Samuelson, L. Lundén and K. Schramm (*Z. anal. Chem.*, 1953, 140 [5], 330-335).—Quant. separation of Ca, Mg, Sr and Ba from V, Fe, Al, Cu, Ni and Co in a column filled with anion exchanger Dowex-2 converted to citrate is described. The separation must be performed in acid medium (pH 1.95 to 2.85) to prevent the alkali-earth metals from being retained by the exchanger. The alkali-earth metals are determined quant. in the eluate as sulphates. The max. error of the separation amounts to 0.3 per cent.

S. K. LACHOWICZ

650. Complexometric determination of sulphate and calcium (and possibly magnesium) ions in sodium chloride solutions. P. J. Hol (*Chem. Weekbl.*,

1954, 50 [2], 21-23).—A complexometric method for determining sulphate and Ca (plus Mg, where necessary) in NaCl soln. is described; the accuracy is  $\pm 0.3$  to 0.4 per cent. Use is made of the previously described method (*Anal. Abstr.*, 1954, 1, 30) of "under-exposure" of the solutions to be titrated. In contrast to the colour change from red to blue, produced by the Eriochrome Black T indicator, the soln. was titrated to bright green in presence of  $\text{BaSO}_4$ ; this method also gave satisfactory results in determinations of hardness of water.

A. STORFER

651. Fractionation of barium-radium mixtures as chromates by precipitation from homogeneous solution. M. L. Salutsky, J. G. Stites, jun., and A. W. Martin (*Anal. Chem.*, 1953, 25 [11], 1677-1681).—Fractional pptn. from homogeneous soln. was investigated as a means for rapid concn. of Ra from Ra-Ba mixtures. Homogeneous pptn. was attained when  $\text{HNO}_3$  soln. of the chromates were gradually neutralised by  $\text{NH}_3$ , which was generated by the spontaneous hydrolysis of previously added urea or KCNO. The separation was improved by decreasing the temp. at which the pptn. were made, but was not affected by the composition of the mixture or the concn. of Ba in the soln. In 19 fractionation steps the composition was changed from a Ba to Ra ratio of 96,930 to 1 to a ratio of 0.18 to 1. The separation per step was better than that by the  $\text{Cl}^-$  or  $\text{NO}_3^-$  fractionation and was equal to that of  $\text{Br}^-$  fractionation.

G. P. COOK

652. Amperometric titrations with potassium ferrocyanide. I. Determination of zinc. B. Khosla and H. C. Gaur (*J. Indian Chem. Soc.*, 1953, 30 [9], 637-640).—The amperometric titrations of oxygen-free (1.5 to 4.5 millimolar) solutions of Zn in 0.1 N KCl medium with standard  $\text{K}_4\text{Fe}(\text{CN})_6$  have been carried out at a dropping-mercury electrode potential of  $-1.18$  V vs. the S.C.E. Gelatin was used as a maximum suppressor. It was found that the pH of the Zn soln. should be  $\leq 2.1$ . The titrations are best done at  $60^\circ\text{C}$ , when the results are within 1 to 2.5 per cent. of the calculated values.

O. M. WHITTON

653. The complexometric determination of zinc in the presence of large quantities of magnesium. E. G. Brown and T. J. Hayes (*Anal. Chim. Acta*, 1953, 9 [5], 408-414).—Zn cannot be determined in the presence of a large proportion (21:1) of Mg by the method previously described (*Brit. Abstr. C*, 1953, 498). Mg is therefore substantially removed by passing the soln. through a column of Amberlite IR-120 cation-exchange resin in the Na form, while the Zn is retained in soln. as its cyanide complex. The Zn complex is then decomposed by adding aq. formaldehyde and the Zn ions are titrated at pH 6.8 with a solution of disodium dihydrogen ethylenediaminetetraacetate.

W. C. JOHNSON

654. The estimation of mercury: action of phenoxycetic acid on salts of mercury. S. K. Datta (*J. Indian Chem. Soc.*, 1953, 30 [9], 657-660).—Methods are described for the use of phenoxycetic acid as a reagent for estimating Hg in mercurous nitrate or perchlorate, and mercuric nitrate or perchlorate solutions, by direct weighing of the salt. By this method Hg can be separated from Ag and Pb and determined in their presence. The mercuric salt of phenoxycetic acid has m.p.  $135^\circ\text{C}$ , and the mercurous salt m.p.  $178^\circ\text{C}$ .

O. M. WHITTON

**655. Analytical chemistry of beryllium.** F. A Vinci (*Anal. Chem.*, 1953, **25** [11], 1580-1585).—The analytical chemistry of Be is reviewed. Methods include detection by *p*-nitrophenylazo-orninol or other colour or fluorescence methods; quant. determination by separating  $\text{Be}(\text{OH})_2$  and igniting to  $\text{BeO}$ ; precipitation of ammonium beryllium phosphate, ignition and weighing as  $\text{Be}_3\text{P}_2\text{O}_7$ ; and conversion of  $\text{Be}(\text{OH})_2$  to weakly ionised  $\text{BeF}_2$  with consequent release of an equivalent quantity of alkali. Procedures for determining Be metal are tabulated. Methods for analysing ores and alloys of beryllium are summarised. Possible toxicological effects and precautions necessary to combat them are indicated. 56 references. O. M. WHITTON

**656. Sample preparation in the routine spectrographic analysis of aluminium and its alloys.** F. Rohner (*Spectrochim. Acta*, 1953, **5** [6], 473-475).—The relative error component for Al resulting from lack of homogeneity in the sample is often substantially more than  $\pm 1$  per cent. In casting samples, high mould temp. gave inaccurate analyses. A mould is described which is machined from an Al alloy and is fitted with integrally cast cooling fins. Improved accuracy is attained. N. E.

**657. Separation of microgram quantities of aluminium and [ferrie] iron.** H. Teicher and L. Gordon (*Anal. Chim. Acta*, 1953, **9** [6], 507-512).—The method previously described (*Brit. Abstr. C*, 1951, 451) is modified in certain details for microgram amounts of Fe and Al. The method has been used for the separation of up to 200  $\mu\text{g}$  of Fe from comparable amounts of Al in caustic liquors before spectrophotometric determination of Al.

W. C. JOHNSON

**658. Quantitative estimation of gallium in aluminium alloys.** E. Gastinger (*Z. anal. Chem.*, 1953, **140** [4], 252-255).—Ga may be separated together with  $\text{Fe}^{\text{III}}$  from Al and  $\text{Fe}^{\text{II}}$  by extraction with ether saturated with HCl when the HCl is 5.5 to 6.5 *N* in both aq. and ether phases. Twenty-five grams of alloy are carefully dissolved in the calculated quantity of 6 *N* HCl and 50 ml of conc. HCl are added. The soln. is set aside for 1 hr. on a water-bath in an atmosphere of N to prevent oxidation of  $\text{Fe}^{\text{II}}$ . As the soln. is difficult to filter, it is centrifuged and the residue is washed with 0.1 *N* HCl. The filtrate and washings are then made up to 500 ml in a calibrated flask. In order to make the acidity up to 6 *N*, a 5-ml aliquot is withdrawn, and a soln. containing 5 g of NaF, 1 g of K oxalate and 20 g of NaCl in 200 ml of  $\text{H}_2\text{O}$  is added. After stirring, a white ppt. is formed. After allowing the ppt. to settle, the acidity is found by titration with N NaOH and phenolphthalein indicator. A 200-ml aliquot is acidified with enough conc. HCl to make its acidity 5.5 to 6.5 *N*. The soln. is shaken first with 200 ml and then twice with 50 ml of ether previously shaken with an equal vol. of 6.5 *N* HCl. After distillation of the ether from the non-aqueous fraction, a few drops of  $\text{H}_2\text{O}_2$  are added and the residue is dried over a water-bath. A few drops of conc.  $\text{H}_2\text{SO}_4$  are added and the HCl is evaporated over an air-bath. The residue is dissolved in 100 ml of 2 *N*  $\text{H}_2\text{SO}_4$  and any Cu is removed by electrolysis. Ga and any Fe are pptd. by a 5 per cent. cupferron soln. and filtered through a hardened filter under weak pressure; the residue is carefully ignited to  $\text{Ga}_2\text{O}_3$  plus  $\text{Fe}_2\text{O}_3$ . After fusion of the oxides with 8 g of NaOH in a silver crucible, the Na gallate is dissolved

in 100 ml of  $\text{H}_2\text{O}$  and the residue of  $\text{Fe}_2\text{O}_3$  is washed with 0.5 *N* NaOH. A few drops of bromothymol blue are added to the filtrate and washings, followed by a small excess of a 3 per cent. soln. of oxine in ammonium acetate soln. After warming to 70° C, 2 *N* HCl is carefully added until the indicator turns yellow. The soln. is set aside for 1 hr. on a water-bath, and the Ga oxinate is filtered through a porcelain filter crucible and washed first with 20 ml of hot  $\text{H}_2\text{O}$  and then with cold  $\text{H}_2\text{O}$  until the washings are colourless. The residue is dried to constant weight at 120° C. J. H. WATON

**659. Quantitative estimation of gallium in the presence of aluminium and zinc by use of cupferron.** E. Gastinger (*Z. anal. Chem.*, 1953, **140** [4], 245-251).—Ga can be estimated quant. by pptn. with cupferron in 0.1 *N*  $\text{H}_2\text{SO}_4$  soln., in the presence of up to 100 times as much Al in 2 *N*  $\text{H}_2\text{SO}_4$  soln. or of excess Zn in *N*  $\text{H}_2\text{SO}_4$  soln. The total vol. of the soln., in which  $\text{SO}_4^{2-}$  should be the only anion, should not exceed 200 ml. The pptn. is carried out by the addition of 5 per cent. aq. cupferron soln., a large excess being avoided, whilst the temp. is kept at < 10° C. The addition of  $(\text{NH}_4)_2\text{SO}_4$  renders the ppt. more easily filterable. The filtration should be carried out under weak suction through a hardened filter. The ppt. is washed with  $\text{H}_2\text{SO}_4$  of the same concn. saturated with cupferron. On igniting the ppt. to  $\text{Ga}_2\text{O}_3$ , care must be taken to avoid loss through reduction to the more volatile  $\text{Ga}_2\text{O}$ . The presence of 0.5 *M* tartaric acid appears to have no effect when the ratio of Ga to tartaric acid is 1:36. J. H. WATON

**660. Separation of cerium from the rare earths by precipitation as iodate from homogeneous solution.** H. H. Willard and S. T'sai Yu (*Anal. Chem.*, 1953, **25** [11], 1754-1756).—The sample (as  $\text{CeO}_2$ ) containing 25 to 100 mg of  $\text{Ce}^{\text{IV}}$  is dissolved in warm dil.  $\text{H}_2\text{SO}_4$  (2 + 1), diluted to about 300 ml with water and made 0.4 to 0.5 *M* with respect to  $\text{HNO}_3$ , and a few drops of 3 per cent.  $\text{H}_2\text{O}_2$  soln. are added to reduce all  $\text{Ce}^{\text{IV}}$ . The excess of  $\text{H}_2\text{O}_2$  is destroyed by boiling, and the soln. is cooled to about 50° C. Three grams of  $\text{NH}_4\text{IO}_3$  are added, then 1 g of ammonium persulphate, and the soln. is stirred and heated to 70° to 80° C. After 1½ or 2 hr., 2 g of the persulphate are added and the soln. is allowed to stand overnight. The ppt. is filtered and washed with 1 per cent.  $\text{HIO}_3$  soln., ignited and weighed as  $\text{CeO}_2$ . If the sample contains elements other than rare earths, the rare earths are separated as the oxalates, ignited to oxides and the procedure is carried out on a soln. of the oxides in warm dil.  $\text{H}_2\text{SO}_4$ . Sr, Ba, Bi, Sn<sup>+++</sup>, Pb, Ag, Hg, Hg<sup>++</sup>, TiO<sup>++</sup>,  $\text{UO}_2^{++}$ , Th, Mn, Fe<sup>+++</sup>, Zn, Cd, Ca, Cu<sup>++</sup>, Ga and In interfere. Up to 30 mg of any rare-earth oxide introduces no appreciable error. G. P. COOK

**661. Colorimetric estimation of small quantities of cerium in uranium.** J. Huré and R. St. James-Schonberg (*Anal. Chim. Acta*, 1953, **9** [5], 415-424).—A method is described for the estimation of Ce in uranates with an error of > 15 per cent. in the range 20 to 50 p.p.m. The presence of Fe, Mn, Cr, V, Ni, Cu and Al is taken into account.  $\text{UO}_2^{++}$  and Fe<sup>+++</sup> are reduced to a sufficiently low concn. by extracting their thiocyanates with ethyl acetate from a soln. made normal with  $\text{HNO}_3$ . The aq. phase is evaporated to dryness, the excess of CNS<sup>-</sup> is destroyed with conc.  $\text{HNO}_3$  and Br and the final residue is taken up in *N*  $\text{H}_2\text{SO}_4$ . After oxidation with ammonium persulphate, the absorption due to



Ce<sup>+++</sup>, MnO<sub>4</sub><sup>-</sup> and other metals is measured at 360 mμ against a comparison soln. in which the Ce<sup>+++</sup> and MnO<sub>4</sub><sup>-</sup> have been reduced to Ce<sup>+++</sup> and Mn<sup>+++</sup> with oxalic acid. The correction for MnO<sub>4</sub><sup>-</sup> is obtained from the absorption of the oxidised solution at 525 mμ where only MnO<sub>4</sub><sup>-</sup> absorbs.

W. C. JOHNSON

**662. Europium determination in rare-earth mixtures.** D. C. Foster and H. E. Kremers (*Anal. Chem.*, 1953, 25 [12], 1921-1922).—A soln. of rare-earth chlorides is passed through a Jones reductor into an excess of FeCl<sub>3</sub>. An amount of FeCl<sub>3</sub> equiv. to that of the EuCl<sub>3</sub> formed by reduction is reduced to FeCl<sub>2</sub>. The FeCl<sub>2</sub> is then titrated with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> with Na diphenylamine-*p*-sulphonate as indicator, H<sub>3</sub>PO<sub>4</sub> being added to prevent premature oxidation of the indicator. There is no interference from other rare earths, e.g., Sm and Yt, which can exist in the divalent state, as they are not reduced by Zn. With rare-earth samples containing 2 to 3 per cent. of Eu<sub>2</sub>O<sub>3</sub>, analyses can be duplicated to a precision within 1 per cent. When checked against pure Eu<sub>2</sub>O<sub>3</sub> (McCoy, *J. Amer. Chem. Soc.*, 1936, 58, 1577) the results were consistently 3.3 to 3.5 per cent. low. The reason for these low results could not be ascertained.

J. M. JACOBS

**663. Spot test for the detection of thallium with rhodamine B.** F. Feigl, V. Gentil and D. Goldstein (*Anal. Chim. Acta*, 1953, 9 [5], 393-399).—A spot test for Tl is described as follows. One drop of neutral or slightly acid Tl<sup>+</sup> soln. is oxidised to Tl<sup>+++</sup> by adding 1 drop of saturated Br soln., the excess of Br is removed by adding 1 drop of 10 per cent. sulphosalicylic acid, 1 drop of a 0.05 per cent. soln. of rhodamine B in conc. HCl is added and the soln. is shaken with 6 to 8 drops of benzene; the benzene layer is coloured red-violet and fluoresces orange-red in u.v. light. The test is sensitive to 0.03 μg of Tl. Sb, Au and Hg interfere and must be removed by boiling the acidified original soln. with copper wire. A concn. of 1 μg of Tl in 500 ml of water can be detected if the Tl is first concentrated by coprecipitation with MnO<sub>2</sub>.

W. C. JOHNSON

**664. Colorimetric determination of carbon in titanium.** M. Codell, G. Norwitz and S. W. Simmons (*Anal. Chim. Acta*, 1953, 9 [6], 555-560).—In the old method of Eggertz for the determination of C in steel the sample is dissolved in HNO<sub>3</sub> and the yellow colour produced by C is taken as a measure of the C present. This method as applied to steel has a number of disadvantages, but when it is applied in modified form to Ti these disadvantages are obviated; e.g., the impurities in commercial Ti are not of such a character or quantity as to mask the colour, and the phase relationships of the Ti-C system are much simpler than those of the Fe-C system. A 1-g sample is dissolved by warming with 20 ml of dil. H<sub>2</sub>SO<sub>4</sub> (1 + 3 v/v) and 10 ml of fluoroboric acid, the latter being prepared by dissolving 130 g of boric acid in 280 ml of ice-cooled 48 to 50 per cent. aq. HF. Five ml of dil. HNO<sub>3</sub> (1 + 1 v/v) are then added and the TiC is dissolved on boiling for 1 min. The soln. is filtered, diluted to 50 ml and compared photometrically at 450 mμ with a reagent blank. Samples whose C contents have been determined by combustion are used for calibration. The method is suitable for C contents ≥ 0.7 per cent.

W. C. JOHNSON

**665. Conductimetric determination of carbon in metals.** J. E. Still, L. A. Dauncey and R. C. Chirnside (*Analyst*, 1954, 79, 4-17).—The sample is

burnt in O in a combustion tube and resistance furnace, the evolved CO<sub>2</sub> being measured by the change in electrical conductance of the absorbing solution of NaOH or Ba(OH)<sub>2</sub>. An improved loading device is described, and a Perspex conductivity cell provides for continuous circulation of the absorbent and for measurement of conductance without stopping the oxygen flow. A special conductivity bridge and a vibration galvanometer give high precision. A variety of samples of metals and alloys weighing from 0.1 to 3 g and containing from 8 per cent. down to 0.01 per cent. or less of C have been satisfactorily analysed.

A. O. JONES

**666. A semi-micro wet combustion method for the determination of carbon.** E. E. Archer (*Analyst*, 1954, 79, 30-33).—In the method described the sample is digested with a wet combustion mixture containing H<sub>2</sub>SO<sub>4</sub>, phosphoric acid, P<sub>2</sub>O<sub>5</sub>, CrO<sub>3</sub> and KIO<sub>4</sub>, the evolved gases, with air, being passed through a heated silica tube containing a silver spiral into an evacuated flask containing Ba(OH)<sub>2</sub> soln. The residual Ba(OH)<sub>2</sub> is neutralised to thymolphthalein and the pptd. BaCO<sub>3</sub> is then titrated with standard HCl with bromophenol blue as indicator. The method is applicable to hydrocarbons, aliphatic carboxylic acids, alcohols, ketones and various sulphur and chlorine compounds and to aq. solutions containing < 0.5 per cent. of carbon.

A. O. JONES

**667. New flux for determination of total carbon in silicon carbide.** P. L. Norton (*Anal. Chem.*, 1953, 25 [11], 1761-1762).—An economical procedure for determining total carbon in silicon carbide is described. It reduces or eliminates errors from incomplete combustion of carbon or from manipulative processes. The flux comprises a mixture of cryolite (Na<sub>3</sub>AlF<sub>6</sub>) and Pb<sub>2</sub>O<sub>3</sub> (20:100 by wt.). The combustion takes 15 min. In applying the method to the analysis of a National Bureau of Standards standard silicon carbide sample containing 29.12 per cent. of C, a combustion temp. of 1700° to 1750° F was used, and the results were on average 0.02 per cent. low. Possible sources of error are discussed.

O. M. WHITTON

**668. The determination of silicon in tungsten and titanium metal powders, carbide sintering alloys, tungstic oxide and tungstates.** B. Bagshawe and R. J. Truman (*Analyst*, 1954, 79, 17-23).—A method is described for determination of Si in W- and Ti-based compounds and materials and mixtures of them with molybdenum carbide, Ta and Co. After ignition of the material to oxide, it is fused with Na<sub>2</sub>CO<sub>3</sub> and the acidified extract is treated with KMnO<sub>4</sub> soln., the excess of which is removed with FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. After treatment of the soln. with ammonium molybdate, the molybdosilicic acid is reduced to molybdenum-blue by means of ammonium oxalate and FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and the colour is measured absorptometrically.

A. O. JONES

**669. Mass spectrometric analysis of germanium.** R. E. Honig (*Anal. Chem.*, 1953, 25 [10], 1530-1535).—As little as 10<sup>-1</sup> M of impurity in commercial Ge is determined semi-quantitatively by slow stepwise vaporisation at 800° to 1460° K of ≈ 5 mg of sample (obtained from a two-stage enrichment of Ge ingots in a gradient furnace) in an Al<sub>2</sub>O<sub>3</sub> crucible (lined with C or SiO<sub>2</sub>) placed inside a 180° mass-spectrometer tube of 4 in. radius. The beam of neutral particles is ionised by a stream

of low-energy electrons (45 V) and subjected to mass analysis; the resolving power is  $\approx 1/200$ . The impurities separate from the bulk Ge according to their diffusion coefficient in the solid, the respective mean mol. concn. and vapourising temp. being: As  $10^{-5}$  to  $10^{-3}$  (850° to 1160° K), Pb  $10^{-4}$  (800° to 1180° K), Sn  $10^{-4}$  to  $10^{-3}$  (1230° to 1360° K), Fe  $10^{-3}$  (1340° to 1345° K), arsenic phosphides and antimonides, each  $5 \times 10^{-4}$  (850° to 1100° K). The low concn. of Cd cannot be evaluated reliably; *n*-type impurities are determined by the resistivity method. After the sample is completely vapourised, a time integration of ion current intensities yields concn. values that are limited in accuracy mainly by errors in the ion cross-sections and partly by vapourisation of Ge commencing at 1120° K. The data indicate that Ge, Sn and Pb evaporate partly in the mol. state (including Ge, to Ge<sub>2</sub> ion-clusters), whilst As vapourises mainly as As<sub>2</sub> and As<sub>3</sub>. In a suitably designed mass-spectrometer tube it should be possible to determine concn. as low as  $10^{-8}$  M by rapid vaporisation of the specific impurity at a pre-determined temp. above the m.p. of Ge.

W. J. BAKER

**670. Volatilisation of germanium during the ashing of coal.** K. V. Aubrey and K. W. Payne (*Fuel*, 1954, **33** [1], 20-25).—By a spectrographic method of estimation it is shown that there is no significant loss of Ge from coal on ashing at 400° C, but there is a loss of up to 20 per cent. when the coal is placed directly in a furnace at 900° C. Addition of 2 per cent. of NaCl does not reduce the loss.

A. B. DENSHAM

**671. Rapid interferometric analysis of germane-hydrogen mixtures.** M. Green and P. H. Robinson (*Anal. Chem.*, 1953, **25** [12], 1913-1914).—The method depends upon the measurement of the difference in  $n_D$  between H and the H-GeH<sub>4</sub> mixture made by a Rayleigh-Zeiss interferometer fitted with a 500-mm double-chamber gas cell. The method has been used for determining the amount of GeH<sub>4</sub> in the H evolved at the cathode during the electrolysis of solutions of Ge compounds. The accuracy of the method (by using the calibration procedure described) is  $\pm 0.059$  mol. per cent. of GeH<sub>4</sub> and the range is 0 to 15 per cent. of GeH<sub>4</sub>.

J. M. JACOBS

**672. Determination of zirconium in aluminium alloys using *p*-bromo- or *p*-chloromandelic acid.** R. A. Papucci, D. M. Fleishman and J. J. Klingenberg (*Anal. Chem.*, 1953, **25** [12], 1758-1760).—Two procedures for determining Zr in Al alloys in the presence of Cu, Mg, Si, Fe, Mn, Ni, Sn, Sb, Cr, Ti and V are described. The first procedure is applicable to alloys containing less than 0.75 per cent. of Si, and involves direct precipitation of Zr with the reagents *p*-chloro- or *p*-bromomandelic acid. The second procedure is used for alloys containing more than 0.75 per cent. of Si and involves a preliminary separation of Zr as the hydroxide. A procedure for the simultaneous determination of Si and Zr in the same sample is also described.

O. M. WHITTON

**673. Solvent extraction separations of zirconium and niobium.** E. M. Scadden and W. E. Ballou (*Anal. Chem.*, 1953, **25** [11], 1602-1604).—An investigation has been made of various solvent systems for extracting both macro and carrier-free concentrations of Zr and Nb from each other and from all other fission-product elements. A mixture of butyl phosphoric esters in *n*-butyl ether was

found effective. Application of the solvent system to other separations is indicated. Conditions for simply separating Y-group rare-earths from those of the La group with 0.6 M di-*n*-butyl hydrogen phosphate in *n*-butyl ether have been found.

O. M. WHITTON

**674. Spectrographic determination of impurities in zirconium and hafnium.** N. E. Gordon, jun., and R. M. Jacobs (*Anal. Chem.*, 1953, **25** [11], 1605-1608).—A spectrochemical method is presented for determining Al, B, Ca, Cd, Co, Cr, Cu, Fe, Hf, Li, Mg, Mn, Mo, Ni, P, Pb, Si, Sn, Ti, V, W and Zr in Zr and Hf metals and oxides. A sample of metal filings or pulverised oxides is volatilised from cratered graphite electrodes in either an a.c. or d.c. arc, depending on the elements to be determined; the resulting spectra are photographed and evaluated by conventional techniques. Ten p.p.m. of most of the impurities can be determined with an accuracy of  $\pm 30$  per cent.

O. M. WHITTON

**675. Polarography of zirconium salts in methanol.** E. L. Collichman and W. H. Ludewig (*Anal. Chem.*, 1953, **25** [12], 1909-1910).—Some of the difficulties encountered in polarography of aq. solutions are obviated by the use of methanol, and the well-defined reproducible waves that are obtained are not complicated by the simultaneous appearance of hydrogen decomposition waves. The results of slope analysis for Zr(SO<sub>4</sub>)<sub>2</sub> are in the range 0.12 to 0.16 and indicate an irreversible reduction. Two waves are reported for Zr(SO<sub>4</sub>)<sub>2</sub> and are attributed to two different quadrivalent species of Zr in solution because the total  $i_d/C$  values are constant over a tenfold concn. range. The results indicate the possible application of the polarographic method to the analysis of hydrated Zr<sup>IV</sup> salts such as Zr(SO<sub>4</sub>)<sub>2</sub>·4H<sub>2</sub>O.

J. M. JACOBS

**676. Determination of tin.** Methods of Analysis Committee, B.I.S.R.A. (*J. Iron & Steel Inst.*, 1954, **176** [2], 156-157).—If the sulphide separation of tin from highly alloyed steels as described in B.S. 1121: Part 20: 1951 is omitted, the method can be applied to the determination of tin in iron, carbon steels and low alloy steels (B.S. 1121: Part 7: 1948). If direct reduction and titration are used either sulphurous acid must be omitted or the excess of it must be boiled off. Copper and phosphorus do not interfere.

C. J. KEATTCH

**677. Phenylhydroxy-acids as reagents in inorganic analysis. I. Mandelic acid as reagent for lead ions.** A. Waksmundzki and B. Szucki (*Ann. Univ. M. Curie-Skłodowska*, **AA**, 1951, **6**, 63-72).—Results of conductimetric and gravimetric determinations of Pb<sup>++</sup> in 50 per cent. ethanol soln. show that mandelic acid can be used for quant. estimation of Pb<sup>II</sup>. The formula of the ppt. obtained by adding mandelic acid to Pb(NO<sub>3</sub>)<sub>2</sub> has been established as Pb(OOC-CHOH-C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>·Pb(NO<sub>3</sub>)<sub>2</sub>. From Pb acetate soln. mandelic acid precipitates Pb(OOC-CHOH-C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>·Pb(OOC-CH<sub>3</sub>)<sub>2</sub>.

S. K. LACHOWICZ

**678. Titrimetric determination of thorium.** J. J. Fritz and J. J. Ford (*Anal. Chem.*, 1953, **25** [11], 1640-1642).—The method described consists in a direct titration of Th with a standard solution of disodium ethylenediaminetetra-acetate (Versene), a soluble complex being formed. An internal indicator (Alizarin red S) gives a sharp reversible colour change at the end-point. Stoichiometric results are obtained in the pH range 2.3 to 3.4.



The end-point can be determined with an estimated maximum error of  $\pm 0.01$  ml by use of a 10-ml burette or  $\pm 0.05$  ml with a 50-ml burette, Li, Na, K, Mg, Ca, Sr, Ba, La, Cr, Mn, Co, Ag, Zn, Cd or  $\text{UO}_2$  ions do not interfere. However, cations including  $\text{TiO}^{2+}$ ,  $\text{ZrO}^{2+}$ ,  $\text{Ce}^{4+}$ ,  $\text{VO}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Sn}^{4+}$ ,  $\text{Sn}^{2+}$ ,  $\text{Pb}^{2+}$  and  $\text{Bi}^{3+}$  and anions that form precipitates or complexes with thorium such as phosphate, fluoride, oxalate, sulphate and mandelate and large concentrations of acetate do interfere. By extraction with mesityl oxide, Th can be separated from all metals except Zr, U and V before a titration with Versene.

O. M. WHITTON

**679. Extraction of heteropoly acids. Application to determination of phosphorus.** C. Wadelin and M. G. Mellon (*Anal. Chem.*, 1953, **25** [11], 1668-1673).—A study was made of liquid-liquid extraction to evaluate its applicability to separation of heteropoly acids, the efficiency of separation being measured by u.v. spectrophotometry. The acids studied were molybdophosphoric, molybdoarsenic, molybdosilicic and molybdo germanic acids and the solvents were selected to include representatives of the classes: alcohol, ketone, ester, ether, hydrocarbon and chlorinated hydrocarbon. The following procedure is recommended for the determination of P. A sample containing up to 50  $\mu\text{g}$  of P is treated in any way that ensures that the P is present as orthophosphate in 15 ml of water whose pH is between 5 and 9 and that the concn. of other ions is below certain max. values. Ten ml of reagent soln. (1.5 per cent. w/v sodium molybdate dihydrate in a 20 per cent. v/v soln. of HCl of sp. gr. 1.19) is added and the soln. is extracted with 10 ml of *n*-butanol in  $\text{CHCl}_3$  (1 + 4 v/v). The extracts from a second extraction are combined and diluted to 25 ml. The absorbance of the soln. is measured at 310  $\mu\text{m}$  and the P is determined by reference to a calibration graph. A table giving max. permissible amounts for 23 interfering ions and a modification of the above method for determination of P in steel are also given. Steels containing up to 0.1 per cent. of P were successfully analysed in the presence of as much as 1.4 per cent. of Mn, 0.3 per cent. of Si, 1.0 per cent. of Cr or 0.2 per cent. of V.

G. P. COOK

**680. Amperometric determination of phosphoric acid with ferric chloride.** W. Hubicki, K. Wiecek and J. Wysocka (*Ann. Univ. M. Curie-Skłodowska, AA*, 1951, **6**, 169-175).—The small solubility of  $\text{FePO}_4$  in solutions of pH > 2.4 is made use of in amperometric titrations of  $\text{PO}_4^{3-}$  with  $\text{FeCl}_3$  and a rotating platinum-micro-electrode. The potential applied is 0.1 to 0.2 V and the error varies between 0.2 and 2.5 per cent.

S. K. LACHOWICZ

**681. Separation of arsenic, antimony and tin with thioformamide.** A. Musil, E. Gagliardi and K. Reichl (*Z. anal. Chem.*, 1953, **140** [5], 342-349).—The method consists in pptg. As, Sb and Sn sulphides with thioformamide at different concn. of HCl.  $\text{As}_2\text{S}_3$  is pptd. first from hot 6 N HCl soln.; neutralisation with aq.  $\text{NH}_3$  and addition of oxalic acid precipitates antimony sulphide, which is then separated from the filtrate containing Sn in the form of an oxalate complex. The separation takes  $\approx 1$  hr. and the error in determination of each metal is 0.1 to 0.4 per cent. when the original sample contains  $\approx 100$  mg of each element.

S. K. LACHOWICZ

**682. Ultra-violet spectrophotometric determination of antimony as iodo-antimonous acid.** A. Elkind, K. H. Gayer and D. F. Boltz (*Anal. Chem.*, 1953,

**25** [11], 1744-1745).—A sample containing between 0.15 and 1.8 mg of Sb per 100 ml of soln. is made slightly acidic with  $\text{H}_2\text{SO}_4$ . Ten-ml aliquots are mixed with 25 ml of a soln. containing 14 per cent. w/v KI and 1 per cent. w/v ascorbic acid. The soln. is then made up to 50 ml with dil.  $\text{H}_2\text{SO}_4$  (250 ml of conc.  $\text{H}_2\text{SO}_4$  per litre), and the absorbance is measured at 330  $\mu\text{m}$  against a reagent blank as reference. Beer's law is also followed at 425  $\mu\text{m}$ .  $\text{Hg}^{2+}$ ,  $\text{Pb}^{2+}$  and  $\text{BiO}^+$  interfere at the u.v. wavelengths and  $\text{BiO}^+$  mainly at the visible wavelengths. With 2 p.p.m. of Sb the procedure gives a standard deviation of  $\pm 3.3$  per cent. at 330  $\mu\text{m}$  and with 10 p.p.m. of Sb a standard deviation of  $\pm 1.1$  per cent. at 425  $\mu\text{m}$ .

G. P. COOK

**683. Colorimetric determination of niobium in titanium alloys.** G. Norwitz, M. Codell and F. D. Verderame (*Anal. Chim. Acta*, 1953, **9** [6], 561-574).—The literature on the separation and determination of Nb is reviewed (89 references). According to the method proposed the Nb is separated from all but a small fraction of the Ti by dissolving the alloy in a mixture of HF and  $\text{HNO}_3$ , concentrating, adding tannin, dil. HCl and boric acid, boiling, separating the ppt. after leaving it to settle overnight, boiling it again with a soln. of tannin in dil. HCl, setting it aside for 2 hr. and filtering. The ignited ppt. is fused with  $\text{KHSO}_4$  and the melt is dissolved in 1.2 M tartaric acid. The Nb is determined photometrically in this soln. by the thiocyanate method of Freund and Levitt (*Brit. Abstr. C*, 1952, 194) modified by reading at 420  $\mu\text{m}$  to reduce interference from Ta. The method is suitable for alloys containing 0.05 to 10 per cent. of Nb. For a Nb content < 1 per cent. the Ta should be  $\geq 0.5$  per cent.; for alloys containing  $> 1$  per cent. of Nb the Ta to Nb ratio should be  $\geq 2$  to 5. The W content should be  $\geq 1$  per cent. No significant interference arises from other elements that are likely to be present.

W. C. JOHNSON

**684. Studies in metal tellurates: determination of bismuth as bismuth tellurate.** G. S. Deshmukh and E. T. Varkey (*J. Indian Chem. Soc.*, 1953, **30** [9], 645-646).—Results are given for the gravimetric determination of Bi as bismuth tellurate. It is shown that the compound under the given operative conditions has the formula  $(\text{BiO})_2\text{TeO}_4 \cdot 2\text{H}_2\text{O}$ , and that the amount of Bi, calculated on the basis of the formation of this compound, agrees within limits of experimental error with that obtained by the standard pyrogallol method.

O. M. WHITTON

**685. Quantitative determination of oxygen in gases.** L. P. Pepkowitz and E. L. Shirley (*Anal. Chem.*, 1953, **25** [11], 1718-1720).—The method described is essentially the classical  $\text{Mn}(\text{OH})_2$  method of Winkler, carried out in a special apparatus (illustrated), with special precautions taken to eliminate errors from (i) dissolved O in reagent solutions, (ii) air oxidation of iodide to iodine, (iii) presence of  $\text{Mn}^{III}$  in  $\text{Mn}^{II}$  salt (the most serious source of error, overcome by preparing the  $\text{MnSO}_4$  in the apparatus in the presence of excess of Mn), (iv) poor contact between gas and solid, (v) contamination during acidification and (vi) determination of small amounts of I by titration (overcome by extracting the I with toluene and measuring colorimetrically). The method is absolute with a zero blank, the standard deviation for precision is  $\pm 0.77$  p.p.m. and the sensitivity is 0.5 p.p.m. Small samples can be analysed, because the reactions can take place below atmospheric pressure. The

apparatus can be constructed from readily available laboratory equipment and can be made portable.

G. P. COOK

**686. Direct determination of oxygen in less familiar metal oxides.** H. R. Hoekstra and J. J. Katz (*Anal. Chem.*, 1953, **25** [11], 1608-1612).—A procedure is described for quant. determining oxygen in a number of metal and metalloid oxides by treatment with  $\text{BF}_3$  and measurement of the O evolved. The method has an accuracy of  $\pm 0.4$  per cent. for 25 to 100-mg samples and can be also used for analysing some oxide solid solutions, mixed oxide phases, ternary oxides and oxygenated anions, for determining combined oxygen impurities in some metals or metal salts, and possibly for determining the degree of hydration of metal salts. The bromine trifluoride reagent (prep. described) liberates molecular oxygen at  $75^\circ\text{C}$  from oxides of all metals that form volatile fluorides or fluorides soluble in bromine trifluoride. O. M. WHITTON

**687. Application of the evolution procedure to the estimation of sulphur in chromium metal and various ferrochrome alloys.** O. Horak (*Z. anal. Chem.*, 1953, **140** [4], 255-261).—The author's modified evolution procedure for estimating S in cast iron by use of  $\text{H}_3\text{PO}_4$  instead of HCl for opening up the sample (*Brit. Abstr. C*, 1953, 205) is successfully applied to various samples of ferrochrome and chromium metal. The method is equally applicable to Cr-, Cr-Ni- and Ni-steels. The results are reproducible to 0.002 per cent. Ten grams of sample, ground finely enough to pass through a 3-mm sieve, are weighed out into the quartz round-bottomed flask of the previously described distillation apparatus and dissolved in 60 per cent. pure S-free  $\text{H}_3\text{PO}_4$ . A stream of  $\text{CO}_2$  is passed through at a rate of 2 to 3 bubbles per sec., whilst the flask is heated to  $320^\circ$  to  $330^\circ\text{C}$  for  $2\frac{1}{2}$  hr. in an oil-bath, the temp. of  $320^\circ$  to  $330^\circ\text{C}$  being maintained for  $\frac{1}{2}$  hr. The  $\text{H}_2\text{S}$  evolved is determined iodimetrically, preferably by a 0.02 N  $\text{I}^- - \text{IO}_3^-$  mixture instead of 0.02 N  $\text{I}$ . To avoid interference from  $\text{PH}_3$ , it is recommended that the  $\text{CdS}$  be filtered off, and washed with Cd acetate soln. The ppt. and filter are then returned to the receiver, 60 ml of Cd acetate soln. are added and the material is mixed with 25 to 50 ml of  $\text{I}$  soln. according to the S-content. After acidification with 30 ml of conc. HCl, the excess of  $\text{I}$  is titrated. A blank estimation is carried out with a sample of S-free iron or iron of known sulphur-content. The whole operation takes about 3 hr. J. H. WATON

**688. Amperometric determination of selenious acid.** W. Hubicki and M. Dabkowska (*Ann. Univ. M. Curie-Skłodowska, AA*, 1951, **6**, 161-168).—Amperometric titrations of  $\text{H}_2\text{SeO}_3$  with  $\text{H}_2\text{S}(\text{NO}_3)_2$  in presence of a large excess of  $\text{H}_2\text{SO}_4$  or  $\text{Na}_2\text{SO}_4$  and a rotating platinum micro-electrode are described. Best results are obtained by applying a p.d. of 0.05 to 0.2 V. The error of a single determination varies between 0.3 and 2.3 per cent. S. K. LACHOWICZ

**689. Spectrophotometric determination of selenium and tellurium in concentrated sulphuric acid.** S. E. Wiberley, L. G. Bassett, A. M. Burrill and H. Lyng (*Anal. Chem.*, 1953, **25** [11], 1586-1588).—A spectrophotometric method of determining small amounts of Te or Se in Mg alloys is described. After Te or Se is precipitated from soln. by reduction and dissolved in conc.  $\text{H}_2\text{SO}_4$ , the absorbance of the soln. is measured at 350  $\text{m}\mu$  for Se and at 520  $\text{m}\mu$

for Te. In a 1-cm cell, 50  $\mu\text{g}$  of Te or 200  $\mu\text{g}$  of Se per 50 ml of  $\text{H}_2\text{SO}_4$  soln. can be determined. Tellurium in Mg alloys can be successfully determined. Selenium can be determined in the presence of equal amounts of Te, but Te cannot be determined in the presence of equal amounts of Se.

O. M. WHITTON

**690. The absorptiometric determination of tellurium in tellurium-lead alloys.** E. G. Brown (*Analyst*, 1954, **79**, 50-53).—The alloy is dissolved in a mixture of acetic acid and hydrogen peroxide, the soln. is boiled after addition of NaCl and HCl (to form double salts with Pb) and thiourea to form a soluble complex with Cu. Elemental Te is then pptd. by addition of  $\text{SnCl}_2$ , collected in a Gooch crucible, dissolved in  $\text{HNO}_3$  and after neutralisation converted to the reddish-yellow K iodotellurate by addition of KI. Many of the details of the method are critical. Sb (not normally present in Te-Pb alloys) interferes. Possible interference by Se, As and Bi was not studied as the method was devised for simple alloys made to the British Standard Specification 334:1934. The method is rapid and the accuracy is adequate for control purposes.

A. O. JONES

**691. Colorimetric determination of chromium in titanium alloys.** G. Norwitz and M. Codell (*Anal. Chim. Acta*, 1953, **9** [6], 546-554).—The literature on Cr determination is reviewed (53 references) and the following method is proposed for Ti alloys containing 0.02 to 4 per cent. of Cr. One gram of the alloy is dissolved by heating in 25 ml of  $\text{H}_2\text{SO}_4$  and 160 ml of water, and the soln. is diluted to 300 ml, treated with 5 per cent. aq.  $\text{H}_2\text{O}_2$  until the blue colour of  $\text{Ti}^{+++}$  disappears or until a faint titanium peroxide colour appears and then boiled for 1 to 2 min. Twenty ml of 15 per cent. w/v ammonium persulphate and 10 ml of 1 per cent. w/v aq.  $\text{AgNO}_3$  are added, heating is resumed and 3 per cent. w/v aq.  $\text{KMnO}_4$  is added until the soln. is pink and then a few more drops are added in excess. The soln. is boiled for 10 min. and more aq.  $\text{KMnO}_4$  is added if the colour is discharged; it is then boiled for 10 min. with 3 ml of dil. HCl (1 + 3 v/v) and if the  $\text{MnO}_4^-$  colour does not disappear in the first 2 min. 2 ml more of dil. HCl are added. The cooled soln. is diluted to 500 ml and set aside for 30 min. For an alloy containing  $\geq 1$  per cent. of Cr a 5-ml aliquot is taken; for 1 to 2 per cent. of Cr 2 ml and for 2 to 4 per cent. 1 ml are taken. The aliquot is diluted if necessary to 5 ml with dil.  $\text{H}_2\text{SO}_4$  (25 ml per 500 ml), diluted to 40 ml with water, treated with 2.0 ml of a 0.25 per cent. w/v soln. of diphenylcarbazide in 95 per cent. ethanol and diluted to 50 ml. In 10 to 40 min. the soln. is compared photometrically at 580  $\text{m}\mu$  with a blank containing 5 ml of dil.  $\text{H}_2\text{SO}_4$  (25 ml in 250 ml) and 2.0 ml of diphenylcarbazide soln. in 50 ml. A calibration graph is prepared from known amounts of  $\text{K}_2\text{Cr}_2\text{O}_7$ , reduced with  $\text{Na}_2\text{SO}_3$ . None of the elements found in commercial Ti alloys, including Fe, V, Mo and W, interferes with the method. W. C. JOHNSON

**692. Polarographic determination of molybdenum in presence of tungsten.** L. Meites (*Anal. Chem.*, 1953, **25** [11], 1752-1753).—A  $\text{M Na}$  citrate buffer of pH 7.0 when used as a base for  $\text{Mo}^{\text{VI}}$  gives a wave possessing a small max. but an excellently defined plateau,  $\text{W}^{\text{VI}}$  neither precipitating nor giving an interfering wave. The  $E_{\frac{1}{2}}$  value is  $-0.927 \pm 0.002\text{V}$  with respect to the S.C.E. and the slope shows that the reduction is irreversible. The residual current in citrate does not appear to be affected by the

addition of as much as 0.2 M tungstate. The mean error in the determination of 0.05 to 10 mg of Mo<sup>VI</sup> in the presence of 1.1 g of W<sup>VI</sup> corresponds to about  $\pm 2$  per cent. of the amount of Mo<sup>VI</sup> present. G. P. COOK

**693. Analytical chemistry of uranium.** C. J. Rodden (*Anal. Chem.*, 1953, **25** [11], 1598-1601).—Advances in the analytical chemistry of uranium from 1949 to the present are reviewed. In addition to papers on gravimetric, volumetric, colorimetric fluorimetric and radiochemical methods, X-ray absorption and coulometric procedures have been described for the first time. 79 references.

O. M. WHITTON

**694. On the employment of liquid emulsion in the titration of uranium from radioactive minerals.** F. A. G. A. Brandão, E. Frota-Pessoa, N. Margem and W. Perez (*An. Acad. Brasil Cienc.*, 1953, **25** [2], 99-106).—The soln. to be titrated is mixed with liquid nuclear emulsion, and  $\alpha$ -particle tracks from the slide obtained are compared with those from a standard soln. treated under analogous conditions. Examples of the application of the method to analysis of U salt soln. and to determination of the Ra content of uraninite are given. Accuracy is  $\approx \pm 3$  per cent. L. A. O'NEILL

**695. Determination of fluorides.** J. M. Icken and B. M. Blank (*Anal. Chem.*, 1953, **25** [11], 1741-1742).—The method, which is a spectrophotometric adaptation of that used by the A.O.A.C., is based on the bleaching of a Th-alizarin lake by F<sup>-</sup>. The F<sup>-</sup> is isolated from food samples by the usual means of either single or double distillation and the colour reagent is prepared by mixing 10 ml of 0.1 N HCl with 10 ml of 1 per cent. NH<sub>4</sub>OH.HCl, 40 ml of 0.1 per cent. Alizarin red S and 40 ml of 0.05 per cent. Th(NO<sub>3</sub>)<sub>4</sub>.4H<sub>2</sub>O. The distillate is neutralised with KOH to *p*-nitrophenol indicator before addition of the reagent. Measurement is made at 525 m $\mu$  2 hr. after addition of the reagent. The procedure can estimate concn. of F as low as 0.05  $\mu$ g per ml. G. P. COOK

**696. Determination of traces of hydrogen fluoride in inert gases.** D. L. Manning and J. C. White (*Anal. Chem.*, 1953, **25** [11], 1648-1650).—A method is described for determining traces (as little as 10  $\mu$ g) of HF in inert-gas sweepings. The gases are passed through a dil. soln. of boric acid and the increase in conductivity of the soln., which is produced by the reaction between boric acid and HF to form a complex strong acid, is measured. The relationship between conductivity and concentration of HF was linear for up to 1 mg of HF. O. M. WHITTON

**697. The determination of microgram quantities of fluoride. III. The reaction mechanism of the aluminium haematoxylol reagent.** B. J. MacNulty and G. J. Hunter (*Anal. Chim. Acta*, 1953, **9** [5], 425-440).—An account is given of a spectrophotometric study of the reaction between Al<sup>+++</sup> and haematoxylol to form a complex, and of the reaction between the complex and F<sup>-</sup>. It is concluded that the complex is formed not with haematoxylol but with haematein, which is formed from the former by oxidation. This explains the necessity of ageing or oxidising solutions of the dye before complexes will form satisfactorily. The destruction of the complex by F<sup>-</sup> is the result of an alteration in the equilibrium conditions resulting from the addition of F<sup>-</sup> and the formation of an Al hydroxyfluoride containing one atom of F. W. C. JOHNSON

**698. New method for the estimation of chloride in halide mixtures.** C. Mahr and H. Otterbein (*Z. anal. Chem.*, 1953, **140** [4], 261-266).—Br<sup>-</sup> is removed from a Cl<sup>-</sup>-Br<sup>-</sup> mixture by oxidation to Br with Ce(SO<sub>4</sub>)<sub>2</sub> in H<sub>2</sub>SO<sub>4</sub> soln.; the Cl<sup>-</sup> is then estimated by a potentiometric titration with AgNO<sub>3</sub>. CNS<sup>-</sup> and S<sup>2-</sup> can likewise be removed, but I<sup>-</sup> and CN<sup>-</sup> must be eliminated beforehand by another method. The metals Ba, Pb, Mn, Ag, Hg, Cr, Bi and Fe interfere, and Co also if CN<sup>-</sup> is present. Interfering anions are PO<sub>4</sub><sup>'''</sup>, acetate, Fe(CN)<sub>6</sub><sup>'''</sup>, BrO<sub>3</sub><sup>-</sup>, IO<sub>3</sub><sup>-</sup> and ClO<sub>3</sub><sup>-</sup> in the presence of reducing material. CNS<sup>-</sup> interferes when present with Br<sup>-</sup> and I<sup>-</sup>. The approx. neutral soln. of Cl<sup>-</sup> plus other halide ions, with a vol. < 15 ml, is introduced into a special 100-ml distillation apparatus. Twenty per cent. H<sub>2</sub>SO<sub>4</sub> is added, so that the soln. is at least 0.6 N in acid. Ce(SO<sub>4</sub>)<sub>2</sub> is added (0.2 to 0.3 g for every 35 mg of Br<sup>-</sup> and 0.8 to 1.0 g for every 35 mg of CNS<sup>-</sup>). Chlorine-free steam is passed through the soln. (whose vol. must not be > 25 to 30 ml) until the soln. boils and then for 5 min. more. Further Ce(SO<sub>4</sub>)<sub>2</sub> must be added if the yellow colour disappears. After cooling, 1 to 2 ml of 60 per cent. H<sub>2</sub>SO<sub>4</sub> is added and excess of Ce<sup>'''</sup> are destroyed with drops of 3 per cent. H<sub>2</sub>O<sub>2</sub>. The soln. is transferred to a beaker and the Cl<sup>-</sup> is determined potentiometrically. An initial rise in potential will be caused by small amounts of Br<sup>-</sup> not removed by oxidation. If I<sup>-</sup> is present, it is removed as I with H<sub>2</sub>SO<sub>4</sub> and NaNO<sub>2</sub>, and CN<sup>-</sup> if present is removed as HCN or ICN, both before the Ce(SO<sub>4</sub>)<sub>2</sub> oxidation. Should it be necessary to use HNO<sub>3</sub> soln. for the estimation, the acidity must lie between 0.35 and 0.8 N, and a spatula full of Ce(NO<sub>3</sub>)<sub>3</sub> must be added also. The danger of losing Cl is greater than with H<sub>2</sub>SO<sub>4</sub> soln.

J. H. WATON

**699. 5:6-Benzoquinolindimic acid as a colorimetric reagent [for iron].** A. K. Majumdar and B. Sen (*Anal. Chim. Acta*, 1953, **9** [6], 529-535).—5:6-Benzoquinolindimic acid forms with Fe<sup>II</sup> solutions a red ppt. of the composition (C<sub>11</sub>H<sub>6</sub>O<sub>2</sub>N)<sub>2</sub>Fe.3H<sub>2</sub>O. This complex is only slightly sol. in water but is freely sol. in aq. KCN; CN<sup>-</sup> increases the optical density of its solutions. The max. absorption is at 515-520 m $\mu$  and the formation of the complexes provides a colorimetric method for the estimation of Fe with an optimum range of 2.4 to 14 p.p.m. For max. colour development the min. amounts of reagent, KCN and hydroxylamine required are, respectively, 40, 800 and 40 times that of the Fe present. Beer's law is followed and the colour is stable for  $\approx 1$  hr. The temp. should not exceed 35°C. Mg, Ca, Ba and Sr ions form ppt. that can be removed by filtration without affecting the Fe<sup>II</sup> estimation. Ag dissolves in the excess of cyanide. The following ions may be present in the ratios indicated relative to the Fe<sup>II</sup> content: PO<sub>4</sub><sup>'''</sup>, 2; acetate, 20; oxalate, 2; tartrate, 6; WO<sub>4</sub><sup>'''</sup>, 5; Mo<sub>7</sub>O<sub>24</sub><sup>'''</sup>, 50; formate, 2; F<sup>-</sup>, 30; B<sub>3</sub>O<sub>3</sub><sup>-</sup>, 2; citrate, 10; benzoate, 30; VO<sub>3</sub><sup>-</sup>, 20; Mn<sup>++</sup>, 40; Cd<sup>++</sup>, 100; Ni<sup>++</sup>, 30; Co<sup>++</sup>, 8; Th<sup>+++</sup>, 10 (filtered); UO<sub>2</sub><sup>++</sup>, 70; Pb<sup>++</sup>, 10 (filtered); Be<sup>++</sup>, 4 (filtered); Al<sup>+++</sup>, 80 (alkali added). The following have no effect: Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>, SCN<sup>-</sup>, SO<sub>4</sub><sup>''</sup>, NO<sub>3</sub><sup>-</sup>, salicylate or the alkalis.

W. C. JOHNSON

**700.  $\alpha$ -Picolinic acid as a colorimetric reagent [for iron]. IV. A comparative study.** A. K. Majumdar and B. Sen (*Anal. Chim. Acta*, 1953, **9** [6], 536-537).— $\alpha$ -Picolinic acid, previously proposed as a colorimetric reagent for Fe (*Brit. Abstr.*

C, 1953, 499 and 502), has been applied to the estimation of Fe in limestone, quartzite and sand. The results are found to compare favourably with those obtained with *o*-phenanthroline.

W. C. JOHNSON

**701. Simultaneous titration of iron and copper with ethylenediaminetetra-acetic acid. Spectrophotometric end-points.** A. L. Underwood (*Anal. Chem.*, 1953, 25 [12], 1910-1912).—Previous work has shown the feasibility of using photometric end-points in the simultaneous titration of two or more cations with ethylenediaminetetra-acetic acid, provided the stability const. of their complexes with this reagent are large enough and differ sufficiently, and that the spectra of the complexes permit selection of suitable wavelengths. Sweetser *et al.* (*Brit. Abstr. C*, 1953, 306) report good photometric titrations of  $\text{Fe}^{3+}$  and  $\text{Cu}^{2+}$  separately with the reagent. The stability const. of the complexes of these ions are large and differ sufficiently widely for their simultaneous titration, the  $\text{Cu}^{2+}$  complex absorbing strongly at 745  $\text{m}\mu$ , where the  $\text{Fe}^{3+}$  complex does not absorb. Results obtained by the method are reported, together with results of its application to an Al alloy containing small amounts of Cu and Fe. Provided that the bulk of the Al is removed, the troublesome drifting that occurs during the titration of Cu when Al is present is eliminated, and the remaining Al has no appreciable effect on the result. Interference from other cations would be expected to be small because of the large stability const. of the  $\text{Fe}^{3+}$  and  $\text{Cu}^{2+}$  complexes and because any possible interference is minimised by working at a low pH. J. M. JACOBS

**702. The simultaneous spectrophotometric determination of iron and copper using 1:10-phenanthroline.** D. H. Wilkins and G. Frederick Smith (*Anal. Chim. Acta*, 1953, 9 [6], 538-545).—The complete separation of Cu and Fe can be effected by one extraction with *n*-octanol of an aq. solution containing both metals as their 1:10-phenanthroline complexes. The  $\text{Cu}^{2+}$  is then determined spectrophotometrically in the separated organic layer and the  $\text{Fe}^{2+}$  in the aq. layer. The method has been investigated in the range of  $\approx 0.05$  to 0.10 mg of each metal by the following procedure. To 5 ml of the soln. containing the metals are added 1 ml of a soln. of 1:10-phenanthroline in ethanol ( $2 \cdot 10^{-5}$  moles per litre), 2 ml of 5 per cent. aq. hydroxylamine hydrochloride buffered with ammonium or sodium acetate, and 5 ml of a phosphate buffer of pH 8.3. The soln. is shaken for 2 min. with 4 ml of *n*-octanol and allowed to separate. The aq. layer is drawn off, diluted to 25 ml with ethanol and the  $\text{Fe}^{2+}$  is determined photometrically at 510  $\text{m}\mu$ . The *n*-octanol layer is diluted to 10 ml with ethanol and  $\text{Cu}^{2+}$  is determined at 435  $\text{m}\mu$ . The error for Cu is  $\pm 2$  to  $\pm 3$  per cent. and for Fe  $\pm 1.3$  to  $\pm 1.1$  per cent. W. C. JOHNSON

**703. Separation of iron from aluminium by shaking with ether and tetrahydrofuran.** H. Specker and H. Hartkamp (*Z. anal. Chem.*, 1953, 140 [5], 353-355).—Fe and Al in the form of thiocyanate complexes are separated quant. when in relative concn. from 30:1 to 1:30 by shaking with 2 or 3 portions of ether and tetrahydrofuran. Addition of tetrahydrofuran alters appreciably the distribution ratio of Fe complex between ether and aq. HCl soln. without affecting the Al distribution ratio. The whole operation lasts  $\approx 10$  min. and

analytical means fail to detect Fe in the aq. phase after shaking with three portions of the mixed solvent. S. K. LACHOWICZ

**704. Iron, chromium and aluminium phthalate complexes and their application to the determination of the metals. A heterometric study.** M. Bobtelsky and I. Bar-Gadda (*Anal. Chim. Acta*, 1953, 9 [5], 446-454).— $\text{FeCl}_3$ ,  $\text{AlCl}_3$  and  $\text{Cr}(\text{NO}_3)_3$  have been titrated, singly or as mixtures, with K phthalate in dil. ethanolic solutions according to the heterometric procedure previously described (*Anal. Abstr.*, 1954, 1, 29 and 51). The solubility and pptn. phenomena observed indicate that each metal forms a series of complexes with the phthalate radicle. The following are common to all three metals:  $[\text{R}^{\text{III}} \text{Phth}]$ ,  $[\text{R}^{\text{III}} \text{Phth}_2]$  and  $[\text{R}^{\text{III}} \text{Phth}_3]$ , where  $\text{R}^{\text{III}}$  is a trivalent metal ion and Phth is  $\text{C}_6\text{H}_4(\text{COO})_2$ ; but whereas Fe forms an insoluble compound of the empirical composition  $[\text{Fe}^{\text{III}} \text{Phth}]$ , no such compounds are formed by Al and Cr. The probable reason for this difference and its effect on the titration of mixtures are discussed.

W. C. JOHNSON

**705. Determination of iron, aluminium and chromium by heterometric titration with phthalate.** M. Bobtelsky and I. Bar-Gadda (*Anal. Chim. Acta*, 1953, 9 [6], 525-528).—The heterometric method previously described (*Anal. Abstr.*, 1954, 1, 29 and 51) is applied to the micro-titration of  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$  and  $\text{Cr}^{3+}$  in 2 to 10-mg quantities. In a mixture of  $\text{Fe}^{3+}$  and  $\text{Cr}^{3+}$  or of  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$  both metals can be determined by successive titrations.  $\text{Cr}^{3+}$  and  $\text{Al}^{3+}$  when present together cannot be titrated separately but their sum can be determined.

W. C. JOHNSON

**706. Gas analyses of cast iron produced at various foundries.** B. B. Bach. (*J. Res. Development, B.C.I.R.A.*, 1953, 5 [2], 45-70).—Samples of various cast and pig irons have been taken from twelve industrial iron foundries and the oxygen, hydrogen and nitrogen contents have been determined (the oxygen and nitrogen by analytical techniques devised in the B.C.I.R.A. laboratories). The accuracy of the hydrogen figures is questionable. Relevant data such as the type of furnace and furnace lining, melting temperature have also been noted. These facts, along with the analytical figures have been tabulated, and some tentative conclusions have been drawn. C. J. KEATCH

**707. Mercury-cathode electrolysis and its application to steel analysis.** Methods of Analysis Committee, B.I.S.R.A. (*J. Iron & Steel Inst.*, 1954, 176 [1], 29-36).—Optimum conditions have been evolved for the removal, by mercury-cathode electrolysis, of large amounts (10 g) of elements such as Fe, Cr and Ni from alloys having a wide range of compositions. After electrolysis, determinations of trace elements such as Al, Ti, V, Zr, Ca and Mg can be carried out on the soln. The cell assembly is described and various limitations of the method are listed. An appendix on the cleaning of the used mercury is included; it incorporates a diagram of a still for mercury recovery.

C. J. KEATCH

**708. Absorptiometric determination of cobalt in iron and steel.** Methods of Analysis Committee B.I.S.R.A. (*J. Iron & Steel Inst.*, 1954, 176 [1], 63-66).—Cobalt is determined absorptiometrically in a wide variety of alloy steels by nitroso-R salt. Depending on the apparatus employed, either the



absorption is measured at 560-600  $\mu$  or Ilford 606 filters together with H 503 filters are used. The method is essentially that of Haywood and Wood (*J. Soc. Chem. Ind.*, 1943, 62, 37) except that boiling to destroy the complexes of the interfering ions is eliminated by using the above wavelength (or filters) and adjusting the reagent additions to compensate for the presence of certain alloying elements (e.g., Cu and Ni). Satisfactory results have been obtained with steels containing up to 11 per cent. of Co.

C. J. KEATTCH

**709. Present limitations of platinum-group analysis. Introduction to the symposium on the platinum metals.** W. M. MacNevin (*Anal. Chem.*, 1953, 25 [11], 1612-1613).—Present methods of analysis for the Pt group are too slow to fit the needs of radiochemistry. Systematic quant. studies are needed.

O. M. WHITTON

**710. Concentrating and dissolving platinum metals.** F. E. Beamish and W. A. E. McBryde (*Anal. Chem.*, 1953, 25 [11], 1613-1617).—The efficiency of the fire assay for ruthenium, osmium, rhodium and iridium is discussed. Reference is made to the losses caused by cupellation and to methods of quant. corroding platinum metals and their alloys. Data are provided to prove that certain types of fluxes may "slag off" some of the platinum metals. Assay recoveries may be unsatisfactory if ore mixtures are pre-fused.

O. M. WHITTON

**711. Ultra-violet spectrophotometric determination of platinum.** J. J. Kirkland and J. H. Yoe (*Anal. Chim. Acta*, 1953, 9 [5], 441-445).—A max. in the u.v. absorption at 262  $\mu$  is used for the photometric determination of  $\text{PtCl}_4$ . A Beckman DU spectrophotometer with a 1-cm cell is used and the soln. is buffered at pH 5 with an acetic acid - Na acetate or HCl - Na acetate buffer. The sensitivity is 0.05 p.p.m. of Pt and the precision is  $\pm 1$  per cent. in the range 3 to 11 p.p.m. The following metals cause an error of  $> 3$  per cent. in the determination of 10 p.p.m. of Pt when present in the following concn. (in p.p.m.): Rh, 0.05; Ru, 0.08; Pd, 0.1; Ir, 0.3; Os, 5. Other transition metal salts interfere. The method differentiates  $\text{PtCl}_4$  from  $\text{PtCl}_2$  as the latter exhibits negligible absorption at 262  $\mu$ .

W. C. JOHNSON

**712. Behaviour of platinum-group metals toward ion-exchange resins.** W. M. MacNevin and W. B. Crummett (*Anal. Chem.*, 1953, 25 [11], 1628-1630).—The chloride complexes of Pd, Pt, Rh and Ir behave as anions and are quant. adsorbed by the anion-exchange resins Amberlite IR-48, Dowex-1 and Dowex-2, but not at all by the cation-exchange resin Amberlite IR-100. The ammine complex of Pd behaves as a cation and is quant. adsorbed by Amberlite IR-100 but not by anion-exchange resins. Thus, Pd has been quant. separated from Ir, Pt and Rh singly and in a mixture, and Rh and Pt from Ir. Only 95 per cent. separation of Rh and Pt has been effected. An adaptation of the Gilchrist - Wichers scheme for separating Pd from a mixture with Rh and Ir is suggested. The Rh and Ir may then be separated electrolytically.

O. M. WHITTON

**713. Chemical methods for separating and determining metals contained in platiniferous materials.** R. Gilchrist (*Anal. Chem.*, 1953, 25 [11], 1617-1621).—Traditional methods for analysing platinum are reviewed, and the application of controlled hydrolytic precipitation to the separation of platinum metals from each other, of platinum

metals from the base metals, and of base metals from one another is discussed.

25 references.  
O. M. WHITTON

**714. Reactions involving coloured complexes of the platinum metals.** G. H. Ayres (*Anal. Chem.*, 1953, 25 [11], 1622-1627).—The basis is outlined of continuous-variation, slope-ratio and mole-ratio spectrophotometric methods for determining the reaction stoichiometry in the formation of coloured products from solutions of the platinum metals. The methods have been used to determine the reaction ratios of the reactions of NaCl with  $\text{PdCl}_4$ , of thiocyanate with osmium, of hypochlorite with  $\text{Rh}^{III}$ , and of  $\text{SnCl}_4$  with  $\text{Pt}^{IV}$ . The results are given and discussed. In the last of these reactions, a cationic complex,  $[\text{PtSn}_4\text{Cl}_4]^{++}$  was deduced on the basis of precipitation tests with strong alkali, with phenylarsonic acid and with  $\text{AgNO}_3$ , as well as on spectrophotometric evidence; this complex contains platinum in zero oxidation state.

O. M. WHITTON

**715. Mass spectrometric determination of gas impurities separable by condensation.** A. S. Newton (*Anal. Chem.*, 1953, 25 [11], 1746-1749).—A method of utilising an internal standard in the mass spectrometric determination of traces of non-condensable impurities in condensable gases or condensable impurities in non-condensable gases is presented. The preparation and analysis of synthetic mixtures of gases containing 300 to 2000 p.p.m. of impurities are described. For non-condensable impurities, Ne is used as the standard whereas for condensable impurities a wide range of standards may be used. The accuracy of the method for non-condensable and condensable impurities in the range quoted above is generally within  $\pm 5$  per cent.

G. P. COOK

**716. Simple method for differentiating between borosilicate [glass] and soft glass.** M. K. Carron and R. E. Stevens (*Anal. Chem.*, 1953, 25 [11], 1782).—The end of a glass sample is abraded in a small drop of water on a streak plate or other unglazed porcelain surface impregnated with phenolphthalein indicator. Soft glass almost immediately produces red streaks on the plate, whereas borosilicate glass does not. The test is also a means of indicating the pH that results from the solution of various natural silicate minerals when indicators covering various pH ranges are applied.

G. P. COOK

**717. The use of isotope dilution in determination of geological age of minerals.** R. H. Tomlinson and A. K. Das Gupta (*Canad. J. Chem.*, 1953, 31 [10], 909-914).—A method is described for estimating radioactive  $^{87}\text{Rb}$  and radiogenic  $^{87}\text{Sr}$  by the use of the mass spectrometer and the isotope-dilution technique. Rb (0.01 per cent.) and Sr (0.0001 per cent.) have been determined in 100 mg of mineral. No chemical separation from the mineral was required for the Rb, but the Sr was concentrated by ion-exchange methods. From the expression:

$$T(\text{age}) = \frac{\% \text{ } ^{87}\text{Sr}(\text{radiogenic})}{\% \text{ } ^{87}\text{Rb}} \times \frac{1}{\text{decay const. of } ^{87}\text{Rb}}$$

the ages of one lepidolite and two biotite minerals have been determined, and have been compared with values obtained by other methods. With one of the biotites the effect of weathering has been studied.

D. LIFF

See also Abstracts 638, 746, 747, 812, 826, 833, 859, 872.

## 3.—ORGANIC ANALYSIS

**718. Method for serial micro-determinations of organically combined halogen.** S. J. Pirt and E. B. Chain (*Rendiconti Ist. Sup. Sanit.*, 1953, **16**, 363-366).—The substance is heated in a sealed tube with conc.  $\text{HNO}_3$  and  $\text{AgNO}_3$ . The resulting silver halides are dissolved in ammonia (for Cl or Br) or in KCN (for I) and then reduced to the metal with hydrazine (for Cl or Br) or with zinc powder (for I) leaving the halogens in aqueous solution to be determined either by weighing the silver pptd. or by titration of the halogen. P. HAAS

**719. Automatic combustion apparatus for determination of sulphur and halogen.** T. T. White, C. J. Penther, P. C. Tait and F. R. Brooks (*Anal. Chem.*, 1953, **25** [11], 1664-1668).—An automatic quartz-tube apparatus for the determination of S and halogen in organic materials is described. The sample is vaporised in a stream of N and combustion occurs in the high temp. zone of the combustion tube where a stream of O is injected. Control of the heat applied is achieved by means of a thermocouple. With this furnace an accuracy of 99 per cent or more is attained from a variety of materials containing > 5 per cent. of S or halogen. At lower concn., S or halogen can be determined to  $\pm 0.03$  per cent. Details of the furnace and combustion tube assembly are given, and diagrams of the wiring arrangement are included. G. P. COOK

**720. Systematic micro-detection of arsenic, bromine, carbon, chlorine, fluorine, iodine, nitrogen, phosphorus and sulphur in organic compounds by a modified Lassaigne's method.** G. Widmark (*Acta Chem. Scand.*, 1953, **7** [10], 1395-1406).—A rapid method for the elementary micro-analyses of the elements, As, Br, C, Cl, F, I, N, P and S in organic compounds by a modified Lassaigne method (*Compt. Rend.*, 1843, **16**, 387) is described. A 1 to 5-mg sample of the organic compound is heated at 500° to 590° C with Na in a sealed Pyrex-glass ampoule. No special precautions are necessary. Positive results are obtained for all the substances investigated, including (i) easily volatile compounds, (ii) polynitro, azo and diazo compounds and (iii) heterocyclic compounds. Diazo compounds give the weakest reactions. Each element can be detected in the presence of all the other elements, with the exception of phosphorus, which cannot be detected when arsenic is present. I. JONES

**721. Mass spectrometric analyses of some six- and seven-carbon alcohols.** V. A. Yarbrough (*Anal. Chem.*, 1953, **25** [12], 1914-1916).—A method is described for the analysis of mixtures of  $\text{C}_6$  and  $\text{C}_7$  alcohols by means of the latest model of commercial mass spectrometer, which gives close temp. control of the source and permits accurate measurement of pressures in the 0 to 100- $\mu$  range. The samples are introduced into the instrument by means of a special mercury-covered orifice and through this orifice into the pre-leak bulb by means of a special micro-pipette. Tests on synthetic mixtures show that the reproducibility is within  $\pm 5$  per cent. of the value of the contained component, the average deviation of a given component from its known value being 8.2 per cent. The mass patterns of hexan-1-ol, 2-ethylbutan-1-ol, heptan-2-ol, heptan-3-ol and heptan-4-ol are tabulated. J. M. JACOBS

**722. A paper chromatographic test for hydroxymethyl compounds.** H. Zahn and A. Räuchle (*Z. anal. Chem.*, 1953, **138** [5], 357-359).—By means of a solvent mixture of 80 vol. of *n*-propanol and 20 vol. of water a 2 per cent. soln. of the following compounds was subjected to chromatography: di(hydroxymethyl)urea, *o*-hydroxymethylphenol, N-hydroxymethylacetamide and hexa(hydroxymethyl)melamine. The chromatogram was allowed to run for 3 hr., it was then dried in air, sprayed with fuchsin sulphuric acid, held over conc. HCl vapour, and dried at 105° to 110° C. Di(hydroxymethyl)urea and N-hydroxymethylacetamide appeared as sharp red-violet spots and *o*-hydroxymethylphenol as a light-blue spot. R. J. MAGEE

**723. Separation of hydrogen peroxide from organic hydroperoxides. Application to polarographic analysis of mixtures.** W. M. MacNevin and P. F. Urone (*Anal. Chem.*, 1953, **25** [11], 1760-1761).—A method is described of quant. separating mixtures of hydrogen peroxide and certain organic hydroperoxides, including 3-pentyl, *tert*-butyl, 3-cyclohexenyl, cyclohexenyl and 2:5:5-trimethyl-2-hexylhydroperoxides. Polarographic current-voltage curves are measured of the original mixture and after the hydrogen peroxide-Ti<sup>+++</sup> complex has been precipitated with aq.  $\text{NH}_3$ . The decrease in wave-height is due to the precipitation of hydrogen peroxide. The remaining wave is due to the organic hydroperoxide. O. M. WHITTON

**724. Quantitative determination of acetaldehyde in the presence of crotonaldehyde.** E. Sjöström (*Acta Chem. Scand.*, 1953, **7** [10], 1392-1394).—Acetaldehyde is determined quant. in mixtures of acetaldehyde and crotonaldehyde by heating the mixture with  $\text{NaHSO}_3$  soln. and subsequently distilling off the acetaldehyde in the presence of  $\text{NaHCO}_3$ . I. JONES

**725. Simple chromatographic method for the determination of the  $\text{C}_{10}$ - $\text{C}_{20}$  saturated straight-chain fatty acids.** H. J. Nijkamp (*Nature*, 1953, **172**, 1102-1103).—The determination of the  $\text{C}_{15}$ - $\text{C}_{20}$  saturated fatty-acids by a modification of the method for the determination of the  $\text{C}_4$ - $\text{C}_{10}$  acids (Nijkamp, *Brit. Abstr. C*, 1952, 199) is described. For these a longer column is necessary and bromocresol green is replaced by bromothymol blue (pH 6.0 to 7.6) as indicator. I. JONES

**726. A new method of separation [of fatty acids] on the preparative scale based on "counter-current distribution."** M. Verzele (*Bull. Soc. Chim. Belges*, 1953, **62** [10-12], 619-639).—The counter-current distribution method of Craig *et al.* is applied to preparative work and 77 g of a mixture of propionic and butyric acids are separated. A two-part cell is described containing 20 ml of heavy phase, 20 ml of light phase and 20 ml of free space. Positioning of the cell allows equilibrium and separation of the components. The theory of the method is summarised, and it is shown that for continuous separation, new material must be added to the system at a position between the peaks of the distributed material. The number of transfers of the phases required is related to the partition coeff. of the two solutes between the phases. The least number of cells required is calculated from the (calculated) max. overlap between the separated materials. In the separation of propionic and butyric acids, water and isopropyl ether are used as solvents.

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The partition coeff. of butyric acid between the solvents is variable and depends upon concn., but on assuming a value of 3.1 for this and of 0.9 for propionic acid, a close agreement with theory is obtained for 44 transfers. For preparative work, small quantities of the mixture are continually added at the appropriate cell in the train, and, from the theoretical formulae, assuming the partition coeff. of butyric acid to be 4.9, a minimum of 7 cells are required; 21 are used. Addition of 500 mg (calculated so that the concn. nowhere exceeds 1 g per cell) of the mixture are made at cell No. 11, withdrawals of soln. are made at cells No. 1 and 21, and fresh solvent is added (at the opposite end to its withdrawal as soln.) at No. 2 and 20. The separation is effected in 154 transfers (before stopping the addition of new material) in the ratio of 9 of the lower layer to 5 of the upper. The limits of the method are discussed and it is shown that a ratio of the partition coeff. of 1.2 requires 50 cells and 1107 transfers.

E. J. H. BIRCH

**727. Paper chromatography of some alkoxy acids.** J. H. Nair, III (*Anal. Chem.*, 1953, **25** [12], 1912).—The  $R_F$  values, which are not given in the literature, for a number of semi-volatile alkoxy acids are presented. The values are reproducible within 2  $R_F$  units. In several of the solvent systems the  $R_F$  values in an homologous series increase linearly with increasing mol. wt.

J. M. JACOBS

**728. Acetic anhydride.** British Standards Institution (B.S. 2068:1953, 8 pp.).—The acetic anhydride must be clear, colourless, free from suspended matter, and on distillation at 760 mm by the prescribed procedure and the apparatus specified,  $\leq 95$  per cent. v/v must distil at 138.0° to 141.5° C. The ash must be  $\geq 0.02$  per cent. The material must comply with the prescribed limit tests for Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> which are equiv. to  $\approx 0.01$  per cent. of Cl<sup>-</sup> and  $\approx 0.03$  per cent. of SO<sub>4</sub><sup>2-</sup>. The acetic anhydride content determined by the specified method must be  $\leq 97.5$  per cent. J. M. JACOBS

**729. Analytical separation of the methyl esters of the C<sub>12</sub>–C<sub>22</sub> fatty acids by vapour-phase chromatography.** F. R. Cropper and A. Heywood (*Nature*, 1953, **172**, 1101–1102).—A vapour-phase chromatography technique for the separation and measurement of the C<sub>12</sub>–C<sub>22</sub> fatty-acid esters at 230° C on a column of Celite and Dow-Corning high-vacuum silicone grease is described. Dodecanal, tetradecanal and hexadecanal are separated under similar conditions.

I. JONES

**730. The photographic recording of paper chromatograms of carbohydrates and a new spray reagent.** G. L. Kilgour and G. S. Dutton (*Canad. J. Chem.*, 1953, **31** [12], 1260–1261).—A method of preparing permanent photographic records of paper chromatograms with the aid of u.v. light is described. This renders visible spots that would not be noted by spraying and inspection in visible light, and is particularly applicable to uronic and biuronic acids. A 2 per cent. soln. of aniline hydrogen malonate in glacial acetic acid was found to be the best spray reagent for simple sugars, although for general work aniline trichloroacetate soln. was preferable.

D. LIFF

**731. Rapid accurate determination of carbohydrates and other substances with the dichromate heat-of-dilution method.** H. F. Launer and Yoshio Tomimatsu (*Anal. Chem.*, 1953, **25** [11], 1767–

1769).—The dichromate heat-of-dilution technique and an investigation of its scope and limitations as applied to starches, sugars, aliphatics and aromatics are described. The determination takes about 5 min. per weighed specimen. The results indicate that organic substances having all carbon atoms individually attached to oxygen atoms, or aromatics, can be determined with small error in the presence of moisture, ash or other non-oxidisable matter, or as pure components in a binary mixture, assuming complete oxidation of C to CO<sub>2</sub>. On the other hand, substances reproducibly oxidised to CO<sub>2</sub> plus a fairly stable intermediate like acetic acid can be determined approx. by taking this product into account theoretically, or accurately by standardising the reagent against the pure substance.

O. M. WHITTON

**732. The iodimetric determination of mannose and other aldoses.** J. R. Collins (*Anal. Chim. Acta*, 1953, **9** [6], 500–506).—A method has been evolved for the iodimetric determination of aldoses, with particular reference to mannose. Mannose reacts more slowly with hypo-iodite than many other aldoses, and secondary reactions become appreciable as the oxidation approaches completion. Conditions affecting the secondary reactions have been investigated and the following proposed method keeps the extent of these reactions small and constant. *Procedure*—To 0.225 g of hexose add equal vol. of N Na<sub>2</sub>CO<sub>3</sub> and 0.1 N iodine soln. (containing 6 per cent. w/v KI). Keep at 20° C for 30 min. and acidify with 6 N HCl. Titrate the excess of I with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> soln.; the titre should lie between 28 and 32 ml. The I used,  $t$ , is the difference between this value and a blank determination with the sugar. The percentage of aldose,  $p$ , is given by  $t = 0.45 + 0.2445 p$  if mannose predominates, or by  $t = 0.45 + 0.2490 p$  if glucose predominates.

W. C. JOHNSON

**733. Determination of dextran with anthrone.** T. A. Scott, jun. and E. H. Melvin (*Anal. Chem.*, 1953, **25** [11], 1656–1661).—Ten ml of anthrone reagent (0.2 per cent. in conc. H<sub>2</sub>SO<sub>4</sub>) are pipetted into borosilicate-glass test tubes in a water-bath at 10° to 15° C and 5 ml of the sample containing 0.02 to 0.04 mg of dextran per ml are added. The tubes are shaken vigorously, while still immersed, until the contents are thoroughly mixed and are then heated at 90° C for 16 min. After cooling to room temp. the absorbances are measured at 625 m $\mu$  against water. The dextran content is calculated by comparing the absorbance with that of a glucose or dextran standard and a blank treated in the same way as the sample. Fructose quant. interferes and chloride gives high results when present at more than twice the concn. of the dextran. A table of the percentage error produced by 19 different chemicals at various concn. is also given. The standard deviation of the method is  $\pm 0.48$  per cent. at an absorbance level of 0.6. G. P. COOK

**734. Colorimetric determination of tetrachloro-hydroquinone.** S. Dahl (*Anal. Chem.*, 1953, **25** [11], 1724–1727).—The reagent is prepared by mixing 5 ml of benzoyl peroxide soln. (2 per cent. w/v in ethyl acetate) with 2 ml of triethylamine; it is then set aside for 30 min. at room temp. An aliquot of the sample containing  $< 0.3$  mg of tetrachloro-hydroquinone in ethyl acetate soln. is added, and the soln. is set aside for 90 min. in the dark. The volume is made up to 50 ml with ethyl acetate and the absorbance is measured at 610 m $\mu$  within

30 min. The tetrachlorohydroquinone content is calculated from a previously determined calibration graph. The method is applicable to the determination of the fungicide in leather, prior treatment of the leather with ethyl acetate in Soxhlet-type extractors being necessary before a slightly modified finish is applied. Chloranil interferes; quinone and hydroquinone also interfere when present in large quantities. The standard deviation is  $\pm 0.7$  mg for a single measurement, or  $\pm 0.03$  per cent. when based on 2.5 g of leather.

G. P. COOK

**735. Determination of primary fatty amines in amine mixtures. Potentiometric titration techniques.** John E. Jackson (*Anal. Chem.*, 1953, **25** [11], 1764-1765).—A method is described of determining the primary-amine content of a mixture of long-chain fatty amines. The chloroform solutions of the amines are potentiometrically titrated after treatment with salicylaldehyde to neutralise the primary amines. The secondary (plus tertiary) amine content of the amine mixtures is equivalent to the acid used in titrating to the first end-point. Primary amine is then calculated by difference from a separate determination of total base. Some results are compared with theoretical derivations.

O. M. WHITTON

**736. 1-Fluoro-2:4-dinitrobenzene as a quantitative reagent for primary and secondary amines.** F. C. McIntire, L. M. Clements and M. Sproull (*Anal. Chem.*, 1953, **25** [11], 1757-1758).—Aq. amine soln. containing 10 to 100  $\mu$ g of amine, 0.05 ml of 1-fluoro-2:4-dinitrobenzene reagent (0.12 ml in 10 ml of ethanol) and 1 ml of 0.1 M NaHCO<sub>3</sub> are mixed and heated at 60° C for 20 min., thus forming the 2:4-dinitrophenylamine compound. To this is added 0.4 ml of NaOH soln. (0.2 N in dioxan) and heating is continued for 60 min. to convert the excess of reagent to 2:4-dinitrophenol. The soln. is then diluted to 10 ml with water and extracted with 10 ml of cyclohexane to remove the 2:4-dinitrophenylamine, whose absorbance is measured in the organic phase, the wavelength used depending on the amine present. Wavelengths for 18 amines are listed, the absorption max. for primary amines lying between 325 and 335 m $\mu$  and for secondary amines between 350 and 360 m $\mu$ . The sensitivity of the method is of the order of 0.1 micromoles. The method was designed for the quant. determination of D-deoxyephedrine but it is suitable for a number of primary and secondary amines.

G. P. COOK

**737. Paper chromatography of quaternary ammonium bases and related compounds.** H. M. Bregoff, E. Roberts and C. C. Delwiche (*J. Biol. Chem.*, 1953, **205** [2], 565-574).—A paper chromatographic method for the identification of quaternary ammonium bases and related compounds in biological material is described. The solvent systems used are ethanol-ammonia (95 ml of 95 per cent. ethanol + 5 ml of conc. aq. NH<sub>3</sub>) and butanol-acetic acid (100 ml of n-butanol, 30 ml of glacial acetic acid and sufficient water to make the soln. just turbid, viz., 80 to 90 ml). A modified Dragendorff solution (KBiI<sub>4</sub>) was the most generally applicable detection reagent. The acetone solutions of the alkaloid reineckates are suitable for chromatography, as it was found that, on chromatography, the reineckates are split into free alkaloid and the Reinecke reagent, each of which travels to its characteristic position. This decomposition was spontaneous in all instances studied, except for choline reineckate. Pre-treatment of the spots with

an excess of AgNO<sub>3</sub> is necessary to decompose choline reineckate prior to chromatography.

B. VINEY

**738. Determination of Schiff bases by titration in non-aqueous solutions.** S. K. Freeman (*Anal. Chem.*, 1953, **25** [11], 1750-1751).—Three procedures in which the primary difference is the use of different solvents are given. The solvents used are acetic acid, CHCl<sub>3</sub> and methyl cyanide, the titrant used being standard HClO<sub>4</sub> in either dioxan or acetic acid soln. End-points are determined potentiometrically. The method was applied to 10 Schiff bases and the results agree well with those by the nitrogen and dinitrophenylhydrazone methods. By means of two of the procedures, viz., with CHCl<sub>3</sub> or methyl cyanide as solvent and dioxan as solvent for the titrant, amine impurities in concentrations as low as 1 per cent. can also be determined.

G. P. COOK

**739. Ion-exchange separation of ammonium and guanidine salts.** M. Bertrand and J. L. Myers (*Canad. J. Chem.*, 1953, **31** [12], 1252-1256).—Large concentrations of ammonium ions interfere with estimations of guanidine by picrate precipitation. These ions can be quant. separated by the use of the resin Nalcite HCR (Canadian trade name for Dowex-50). A method is described which permits estimation of guanidine in the presence of ten times its weight of ammonium salt.

D. LIFF

**740. Use of ion exchangers for the analysis of reaction mixtures.** The alkaline hydrolysis of cyanoacetamide. Sigvard Wideqvist (*Ark. Kemi.*, 1953, **6** [4], 305-308).—The ion-exchange method for the analysis of reaction mixtures is applied to the alkaline hydrolysis at 25° C of cyanoacetamide which proceeds with a velocity about 250 times as great as that of acetamide. This method, which is highly accurate for slow reactions, gives acceptable values of the velocity constant for this fast reaction. Two alternative analytical procedures are described. In the first, the reaction is stopped by adding a sample to a known excess of acetic acid; the mixture flows through the ion exchanger and the acid (added acetic acid and acid from the hydrolysis) is titrated with standard alkali. In the second procedure, the reaction is not stopped before analysis, but for all samples the time of flow through the ion exchanger is kept constant. Both methods give corresponding values for the velocity constant.

D. BAILEY

**741. Quantitative determination of methyl mercaptan, dimethyl disulphide and dimethyl sulphide in a gas mixture following separation of mercaptan by mercuric cyanide and absorption of disulphide and sulphide in benzene.** W. Segal and R. L. Starkey (*Anal. Chem.*, 1953, **25** [11], 1645-1648).—Methyl mercaptan and alkyl sulphides in a gas are quant. determined by removing them with mercuric cyanide and benzene, respectively. The method is described and its application in determining the volatile sulphur products of microbial decomposition, e.g., of methionine, or in brewing, petroleum products and sewage is indicated. Results presented for methyl mercaptan, dimethyl disulphide and dimethyl sulphide are mostly accurate to within 5 per cent.

O. M. WHITTON

**742. Photometric determination of copper in gasoline.** J. K. Livingstone and N. D. Lawson (*Anal. Chem.*, 1953, **25** [12], 1917-1918).—A rapid photometric method for the quant. determination of Cu in petrol is described which is applicable to

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746. Direct

concn. of 0.025-5 mg of Cu per litre with an accuracy of  $\pm 10$  per cent. A 200-ml sample of petrol is extracted 3 times with 30-ml portions of 0.1 N HCl. Residual colour is removed from the Cu soln. by extraction with 40-ml portions of  $\text{CHCl}_3$  after making them alkaline by addition of 5 ml of conc. aq.  $\text{NH}_3$ ; 10 ml of 0.1 per cent. Na diethyldithiocarbamate are then added, the soln. is extracted twice with 40-ml portions of  $\text{CCl}_4$ , the extracts are diluted with  $\text{CCl}_4$  to 100 ml and the percentage transmittance is determined within 5 min. at a wavelength of 440 m $\mu$ . The concn. range for greatest accuracy is 0.027 to 0.074 mg of Cu in the final 100 ml of  $\text{CCl}_4$  and in this range the relative analysis error is 3 per cent. per 1 per cent. absolute error in transmittancy. The standard deviation of the method is 0.0025 mg of Cu per litre. Sulphur up to at least 0.1 per cent. as dibutyl disulphide, and tetra-ethyl-lead up to at least 5 ml per U.S. gall. do not interfere. Iron naphthenate does not interfere at concn. up to 1 mg per litre, but at 2 mg per litre it gives high results. Antioxidants, deactivators and dyes do not interfere. J. M. JACOBS

**743. Determination of total nitrogen in petroleum distillates by catalytic hydrogenation.** C. Wankat and J. G. Gatsis (*Anal. Chem.*, 1953, **25** [11], 1631-1633).—In a method described for determining total nitrogen in petroleum distillates in the range of 1 to 100 p.p.m., the organic nitrogen is quant. reduced to  $\text{NH}_3$  by high-pressure hydrogenation in an autoclave with a Ni catalyst, and the  $\text{NH}_3$  is absorbed on acidic  $\text{Al}_2\text{O}_3$  pills, which are mixed with the catalyst, and determined by a conventional Kjeldahl distillation. Interference from sulphur is eliminated by proper choice of sample size. O. M. WHITTON

**744. Infra-red determination of total aromatics in naphthas and catalytic reformates boiling between 200° and 400° F.** J. Bomstein (*Anal. Chem.*, 1953, **25** [11], 1770-1772).—The theory, development and procedure of an i.r. determination of total aromatics in hydrocarbon fractions by the use of a spectrogram between 5.00 and 5.85  $\mu$  are given. The analysis overcomes difficulties encountered in several techniques, including acid adsorption, and u.v., Raman and mass spectrometry. It is rapid and reasonably accurate. Limitations, interference and errors are discussed. O. M. WHITTON

**745. Assay of certain organic materials by dichromate oxidation.** N. F. Schulz (*Anal. Chem.*, 1953, **25** [11], 1762-1764).—A rapid method is described of determining the simultaneous adsorption of starches and froth flotation collectors (e.g., dodecylamine acetate) on quartz and on haematite when concentrations of less than 250 p.p.m. must be determined. After heating with dichromate reagent (25 g per litre), the soln. is cooled the conc. phosphoric acid and then excess of 0.05 N ferrous ammonium sulphate are added; 0.1 N  $\text{KMnO}_4$  is used for back titration. The over-all precision of measurement attained for the procedure is about  $\pm 0.02$  ml of the 0.1 N  $\text{KMnO}_4$ ,  $\pm 0.02$  mg of starch or  $\pm 0.01$  mg of dodecylamine acetate. Samples containing more than 0.02 mg of Cl are unsuitable. A colorimetric method is described for determining excess of dichromate after oxidation when the solutions are clear and free from interfering coloured substances. O. M. WHITTON

**746. Determination of trace elements in fuel oils. Direct spectrographic method.** C. W. Key and

G. D. Hoggan (*Anal. Chem.*, 1953, **25** [11], 1673-1676).—The method described is applicable to the determination of V, Na, Ni and Ca in residual fuel oils without prior ashing. A rotating-disc electrode composed of graphite and  $\text{Li}_2\text{CO}_3$  is used to introduce the soln. of fuel oil, internal standard and Ba 2-ethylhexoate buffer into a high-voltage spark discharge. An inert atmosphere is maintained in the arc-spark stand and a single exposure covering 3265 to 5915 Å is made on Type 103-F film. The standards are prepared by adding V oleate and Ni, Na and Ca salts of 2-ethylhexoic acid, dissolved in a refined paraffin, to a fuel oil of known composition. The lower limits of detection for the method are for Ca, Na, Ni and V, 0.4, 0.2, 10 and 2 p.p.m. respectively. The precision and sensitivity are comparable to those attained by other methods. G. P. COOK

**747. The determination of small amounts of chromium in textiles by means of diphenylcarbazide.** A. G. Hamlin (*Shirley Inst. Mem.*, 1952-3, **26** [20], 261-271).—The organic matter is destroyed by wet ashing with  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$  and  $\text{HClO}_4$  or, if the inorganic content is low, by dry ashing in the presence of  $\text{Mg}(\text{NO}_3)_2$ . An acid soln. of the residue is treated with a  $\text{CHCl}_3$  soln. of oxine to remove Cu and Fe. The Cr is oxidised to the hexavalent state by boiling with ceric ammonium sulphate. The cooled soln. is treated with 2 ml of diphenylcarbazide reagent (0.25 per cent. w/v in 50 per cent. aq. acetone) and diluted to 50 ml with water. The optical density of this soln. is measured on an absorptiometer calibrated with standard Cr solutions. Special procedures are described for the removal of Fe, Sn, Ti and Zr with cupferron, and of Sb, Cu and Hg as their sulphides. Variations between different batches of diphenylcarbazide is one of the chief difficulties met in this method. A. M. SPRATT

**748. Estimation of salicylanilide ("Shirlan") in cotton or regenerated cellulose rayons.** Anon. (*Shirley Inst. Test Leaflet*, 1953, No. Chem. 19).—The salicylanilide (I) is extracted from the fabric by a dil. soln. of borax in  $\text{H}_2\text{O}$  saturated with cyclohexanol. At the same time a series of standard solutions of I in cyclohexanol is prepared. Portions of the extract and of each standard soln. are treated with an alcoholic soln. of 2:6-dibromoquinone-chloroimide (II) and the blue indophenol colour formed is absorbed on small discs of bleached cotton cloth. The amount of I in the original sample is estimated by comparing visually the colour of the test disc with the standard series. Qual. detection of I in white material is carried out by moistening with cyclohexanol and spotting with II. For coloured material, the I is first transferred to a piece of bleached cloth. A. M. SPRATT

**749. Rapid accurate determination of cellulose with the dichromate heat-of-dilution method.** H. F. Launer and Yoshio Tomimatsu (*Anal. Chem.*, 1953, **25** [11], 1769-1770).—Results obtained by the heat-of-dilution method and by another method requiring external heating but with identical reagents are compared for identical samples of cellulose, starch and glucose. It is shown that cellulose can be determined rapidly and with slight error, in the presence of moisture and usual inorganic impurities, by applying the theoretical factor 0.01240 g of cellulose per ml of 1.835 N (90.00 g per litre)  $\text{K}_2\text{Cr}_2\text{O}_7$  soln., with the dichromate heat-of-dilution method. See also abstract 732. O. M. WHITTON

**750. The laboratory control of pulp and paper manufacture. III.** J. Grant (*Lab. Practice*, 1954, **3** [1], 14-20).—The determination of optical properties (colour, opacity, gloss and light fastness), penetration and absorbancy tests (sizing tests to determine resistance to penetration by writing ink, oil penetration, air permeability, vapour porosity, etc.), fibre identification (by the Herzberg staining test) and identification of dirt content by microscopical examination are reviewed briefly.

J. M. JACOBS

**751. Estimation of carboxyl, aldehyde and ketone groups in chromium trioxide oxystarches.** A. C. Ellington and C. B. Purves (*Canad. J. Chem.*, 1953, **31** [9], 801-813).—Corn-starch amylose was more readily oxidised than the amylopectin by  $\text{CrO}_3$  in acetic acid-acetic anhydride (a non-swelling system). This difference in reactivity nearly disappeared when samples dried through solvent exchange were oxidised in 0.2 M  $\text{H}_2\text{SO}_4$  (a swelling medium). Carboxyl groups in the oxystarches were estimated by ion exchange with 0.5 N Ca acetate or N NaBr, or by titrating to pH 8.5 with 0.1 N NaOH. Carbonyl groups were determined by estimating the  $\text{NH}_2$  obtained from the hydrolysis of cyanohydrins formed by condensation with excess of NaCN. Aldehyde groups in an oxystarch containing 0.16 M of carboxyl and 0.14 M of carbonyl groups were selectively oxidised with  $\text{HClO}_2$  or alkaline hypo-iodite, or selectively condensed with  $\text{NaHSO}_3$ . All estimations indicated that  $\frac{1}{3}$  of the carbonyl groups were aldehydic occupying the sixth positions in the glucose residues. The cyanohydrin of the oxystarch, when saponified, hydrolysed and reduced with HI, gave the lactone of 4-hydroxy-2-methylhexanoic acid, the recovery of which showed that at least 17 per cent. of the carbonyl groups occurred as 2-oxoglucose residues.

D. LIFF

**752. Determination of polystyrene in styrenated alkyl and styrenated epoxy resins.** M. H. Swann (*Anal. Chem.*, 1953, **25** [11], 1735-1737).—Two procedures for the determination of polystyrene are described. One is applicable to either the alkyl or epoxy resins and the other to the alkyls only, the latter however allow the subsequent determination of the oil acid content. In the method for alkyl resins the phthalic anhydride is removed, by filtration from the benzene soln., after saponification with alcoholic KOH, the anhydride content being determined by this means. The filtrate from this is collected and evaporated to apparent dryness and the polystyrene is filtered after digestion with 87 per cent. aq. methanol. In the method for epoxy and alkyl resins, the sample is heated with KOH pellets at  $110^\circ\text{C}$  for 3 hr. The dried mixture is then heated at  $235^\circ\text{C}$  for 45 min. and stirred at 15 min. intervals. The mixture is then added to 87 per cent. aq. methanol and the polystyrene is determined by filtration and weighing, after digestion. For two samples stated to contain 39 per cent. of styrene one gave 39.4, 39 and 39.1 per cent. and the other 40, 40.1 and 39.9 per cent. A styrenated epoxy resin with styrene content of 33.3 per cent. gave 33.8, 33.5, 34.0 and 33.6 per cent.

G. P. COOK

**753. Polarographic analysis of lead driers.** D. A. Skoog and R. L. Focht (*Anal. Chem.*, 1953, **25** [12], 1922-1924).—Most common lead driers can be rapidly dispersed in 0.1 M solutions of dodecylamine acetate to produce clear solutions that give typical

polarographic waves for Pb, the height of the wave being related directly to the concn. of the Pb in the driers. The results obtained when the method was applied to the analysis of a number of commercial lead driers compared favourably with those given by the procedure of the American Society for Testing Materials (a method involving wet ashing and the gravimetric determination of Pb as  $\text{PbSO}_4$ ), except for Pb resinate, which does not disperse satisfactorily in 0.1 M dodecylamine acetate. The polarographic method is rapid and a single analysis can be completed in 20 min. Of the naphthenates of Fe, Co, Mn, Cu and Zn, only Cu naphthenate gives a satisfactory wave. Work on the polarographic lead driers compared favourably with those given analysis of copper soaps is in progress.

J. M. JACOBS

See also Abstracts 633, 637, 804, 828, 830, 851.

#### 4.—BIOCHEMISTRY INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

**754. Direct micro-determination of sodium, potassium and calcium in a single biological specimen.** G. R. Kingsley and R. R. Schaffert (*Anal. Chem.*, 1953, **25** [11], 1738-1740).—A flame photometric method for the micro-determination of Na, K and Ca in a single biological specimen is given. A photomultiplier tube attachment to a Beckman DU flame photometer permits the selection of narrow slit widths of light. The  $\lambda$  settings for the Na, K and Ca determinations are 590, 770 and  $422.7\text{ m}\mu$ , respectively. Good recoveries are attained from specimens of serum and urine; 96.7 to 102 per cent., 98.4 to 102.5 per cent., and 98.9 to 101.9 per cent. being obtained for Na, K and Ca, respectively, in serum. Similar average recoveries, with a slightly greater range, are attained from serum and urine containing extreme quantities of cholesterol, phosphorus, glucose and urea.

G. P. COOK

**755. Measurement of serum cholinesterase activity.** J. G. Reinhold, L. G. Tourigny and V. L. Yonan (*Amer. J. Clin. Path.*, 1953, **23**, 645-653).—Michel's method for assay of cholinesterase was modified by use of an indicator (phenol red) and colorimeter in the determination of pH; this was found as accurate as the original electrometric method and more adaptable to field conditions. Mean figures for blood donors of 0.926 Michel units for white males, 0.814 for negro males, 0.822 for white females and 0.768 for negro females were obtained, with a standard deviation of  $\approx 0.14$  for each.

B. LENNOX

**756. The determination of cholinesterase activity in blood samples absorbed on filter-paper.** K.-B. Augustinsson and G. Heimbürger (*Acta Physiol. Scand.*, 1953, **30** [1], 45-54).—A method is described for the determination of cholinesterase of blood absorbed in small amounts of filter-paper. By use of acetyl- $\beta$ -methylcholine and butyrylcholine as substrates both the erythrocyte acetylcholinesterase activity and the plasma butyrylcholinesterase are determined in two separate drops (0.1 ml) of blood without separating plasma and red cells. A comparison of the values obtained in this way, for the two activities, with those obtained with plasma and cells separated and run against acetylcholine in the



usual way, shows satisfactory agreement. The blood absorbed on the paper and air-dried may be kept at room temp. for a week, or in a refrigerator for several weeks, without any appreciable loss of esterase activity. D. BAILEY

**757. Microbiological thiamine determination in blood by means of an *E. Coli* mutant.** J. D. Jansen and G. J. E. Thyse (*Chem. Weekbl.*, 1954, **50** [2], 18).—A microbiological thiamine assay on 0.2 ml of whole blood is described, with an *E. coli* mutant as test organism. The use of 6 ml of medium and the test-tube technique are involved. A. STORFER

**758. A chemical method for the determination of free choline in plasma.** H. D. Appleton, B. N. La Du, jun., B. B. Levy, J. M. Steele and B. B. Brodie (*J. Biol. Chem.*, 1953, **205** [2], 803-813).—A sensitive and specific method for the estimation of free choline in plasma is described. The method involves extraction of the choline into acetone, evaporation of the acetone and removal of interfering substances from the aqueous residue by butanol and isobutanol extraction. Choline is pptd. as a periodide which is dissolved in ethylene dichloride and measured spectrophotometrically at 365 m $\mu$ . Iodide adsorbed on the periodide ppt. does not interfere in the measurement, and as little as 5  $\mu$ g of choline can be determined. The free-choline level in plasma in normal male adults averages about 4.4  $\mu$ g per ml. Analyses made over a period of several months indicate that each individual maintains a relatively constant plasma level and that this level is not increased after meals or by the oral administration of choline. Evidence is presented suggesting that the level of free choline in plasma is controlled by a process involving metabolic transformation. B. VINEY

**759. The determination of iron and copper in single serum samples.** S. Ventura and J. C. White (*Analyst*, 1954, **79**, 39-42).—By the method proposed Fe and Cu are determined spectrophotometrically in single samples of serum. The metallic ions are liberated from protein by 6 N HCl and the protein is then removed by means of trichloroacetic acid. After reduction of Fe<sup>+++</sup> to Fe<sup>++</sup> with hydroquinone dissolved in dil. aq. ascorbic acid, the test soln. is treated with 2:2'-dipyridyl soln. and then with Na diethyldithiocarbamate soln. The Cu complex is extracted with a mixture of ether and pentanol and the Fe complex remains in the aq. layer. The optical density of the Cu complex is measured at 440 m $\mu$  and that of the Fe complex at 520 m $\mu$ ; the concn. of the complexes are ascertained from standard soln. treated similarly. A. O. JONES

**760. Convenient method for determining chlorides in urine: modification of Northrop's potentiometric titration.** R. H. Kellogg, W. R. Burack and K. J. Isserbacher (*Proc. Soc. Exp. Biol. Med.*, 1952, **81**, 333-338).—The method described is based on the established principles of potentiometric titration and consists in the titration of the chloride with a standard soln. of AgNO<sub>3</sub> in an appropriate electrolyte; the end-point is denoted by the change of voltage at a silver electrode. W. A. GROOM

**761. An improved copper reduction method for the micro-determination of reducing sugars.** H. G. Wager (*Analyst*, 1954, **79**, 34-38).—Certain errors inherent in Nelson's method (*J. Biol. Chem.*, 1944, **153**, 375) have been eliminated by carrying out the

whole test under oxygen-free conditions. The extinction coeff. of the final molybdenum-blue soln. is recorded at a standard time after addition of the molybdo-arsenate reagent. A convenient apparatus for the method is described. A. O. JONES

**762. Determination of reducing sugars. II and III. Criticism of the Blom and Rosted modification of the Lane and Eynon method.** Y. Matsuo and H. Matsui (*J. Ferment. Technol., Japan*, 1953, **31**, 109-111 and 135-139; *Sugar Ind. Abstr.*, 1953, **15** [7], 520 and 521).—II. A glucose table is given for use with the Blom and Rosted method (*Acta Chem. Scand.*, 1947, **1**, 382). III. The Blom and Rosted method (*loc. cit.*), which is not affected by NaCl, gives high values in presence of acid hydrolysate of soya-bean protein. A Bertrand micro-method gives low results and the end-point in the Lane and Eynon method is indistinct. Cystine interferes. The Blom and Rosted method is excellent for the determination of soya sauce. SUGAR IND. ABSTR.

**763. Determination of reducing sugars. IV. Modified Blom's method.** Y. Matsuo and H. Matsui (*J. Ferment. Technol., Japan*, 1953, **31**, 298-300; *Sugar Ind. Abstr.*, 1953, **15** [10], 726).—A device is described by means of which improved accuracy is attained by bubbling steam (by way of a tube sealed into the titration vessel) through the reaction mixture during titration. The (open) neck of the vessel is held by a rubber stopper closing a flask in which water is boiled; the steam escapes through the side-tube into the titration vessel. The range of the method is 60 to 160 mg of glucose per 100 ml, with an accuracy of  $\pm 1.6$  per cent. The sugar concn. is calculated (without tables) by the equation:  $Sugar (mg \text{ per } 100 \text{ ml}) = F/\text{titre in ml, where } F = 1541 \text{ for glucose.}$  P. S. ARUP

**764. Copper-ethylenediaminetetra-acetic acid complex in alkaline solution. Qualitative comparisons with Benedict's reagent.** H. Wagerich and B. Harrow (*Anal. Chem.*, 1953, **25** [12], 1925-1926).—Benedict's qual. reagent and the copper-ethylenediaminetetra-acetic acid complex (I) give positive tests (yellow to reddish-brown ppt.) with glucose, galactose, maltose, lactose, fructose, arabinose, xylose and ascorbic acid. The following compounds do not reduce either reagent: sucrose, raffinose, starch, urea, formaldehyde, recryst. egg or bovine albumin, trypsin, casein, creatinine, gelatin, cellulose or thrombin. With CHCl<sub>3</sub> and chloral hydrate, Benedict's reagent gives a slight positive reaction. The reagent containing I and Na<sub>2</sub>CO<sub>3</sub> gives no ppt. with either of these compounds, but when I contains NaOH it gives a positive test with CHCl<sub>3</sub>, although not the chloral hydrate. The optimum pH for the reduction of glucose by I containing Na<sub>2</sub>CO<sub>3</sub> is 10.5; for the reagent containing NaOH it is 10.3 to 12.8, with only a slight change in sensitivity at pH 11.5 to 12.5. Benedict's qual. reagent gives a positive test with 0.1 to 0.2 per cent. of glucose in urine, whereas the reagent prepared from I and Na<sub>2</sub>CO<sub>3</sub> gives a definite yellow ppt. only when at least 0.5 per cent. of glucose is present in the urine. J. M. JACOBS

**765. The separation of sugars and of sugar phosphates by gradient elution from ion-exchange columns.** C. W. Parr (*Biochem. J.*, 1954, **56** [1], xxvii-xxviii).—Mixtures of sugars and sugar phosphates are adsorbed on a column of De-acidite FF prepared in the borate form. Elution is carried out with 0.1 M H<sub>2</sub>BO<sub>3</sub> containing NaCl; a simple device

is described which causes the NaCl concn. to rise to 0.25 M during elution. Fractions are located by the anthrone method and by other methods.

C. E. SEARLE

**766. Paper ionophoresis of phosphoric esters.** M. W. Neil and D. G. Walker (*Biochem. J.*, 1954, **56** [1], xxvii).—Mixtures of inorganic phosphate and triose and hexose phosphates are separated by ionophoresis on paper in various buffers. Complete separation of esters is not always obtained but the method gives useful fractionations of tissue extracts without preliminary treatment. C. E. SEARLE

**767. Photometric micro method for the estimation of acetic acid in urine.** A. Fennesu (*Biochem. Z.*, 1953, **324** [7], 512-516).—The following method permits the estimation of 0.1 to 1.0 mg of acetic acid in small vol. of urine with an accuracy of  $\pm 10$  per cent. Ten ml of urine or a suitable dilution thereof are made alkaline to litmus with solid NaOH; 0.5 g of  $\text{CuSO}_4$  is added and dissolved by shaking. After setting aside in an ice-chest for at least 12 hr. the mixture is centrifuged. To 5 ml of supernatant soln. is added 5 ml of distilled water, 2.5 ml of saturated  $\text{CuSO}_4$  soln. and 4 ml of 15 per cent. milk of lime. After shaking and centrifuging, the fluid is transferred to a 100-ml Kjeldahl flask, the precipitate being washed and the washings also being transferred. The mixture is heated to dryness *in vacuo*. The residue is treated with 15 ml of methanol and then with 3 ml of conc.  $\text{H}_2\text{SO}_4$ , the flask being cooled during the addition of the acid. The liquid is then distilled into a 50-ml Kjeldahl flask containing 4 ml of methanol, the end of the condenser being below the surface of the methanol. After 4 ml of distillate has been collected distillation is stopped, the end of the condenser being washed with 4 ml of methanol, which is added to the 8 ml in the flask. This mixture is redistilled and the process is repeated three times, the product of the last distillation being collected in 4 ml of 1.25 per cent. NaOH instead of methanol. After stirring, the mixture is set aside for 30 min. and the methanol and water are removed by heating in a water-bath. The residue is refluxed with 7 ml of 0.05 N  $\text{K}_2\text{Cr}_2\text{O}_7$  and 7 ml of conc.  $\text{H}_2\text{SO}_4$  for 30 min., the top of the reflux condenser being covered. This treatment oxidises substances other than acetic acid that may have been carried over in the distillation process. After cooling, 4 ml of the soln. is transferred to a centrifuge tube and diluted with distilled water. After addition of 2 drops of saturated  $\text{AgNO}_3$  soln. and 3 ml of conc.  $\text{H}_2\text{SO}_4$  to the liquid in the reflux apparatus, heating is continued for 1 hr. This addition of  $\text{AgNO}_3$  and the increased concn. of  $\text{H}_2\text{SO}_4$  permits the oxidation of the acetic acid. After cooling, a 4-ml sample is diluted with 6 ml of water in a centrifuge tube. Blank solutions should also be run for the above procedures. After removal of the light ppt. by centrifugation the extent of the reduction of the dichromate is determined in a spectrophotometer at 450 m $\mu$ . After correction for the blank and non-specific oxidation, the acetic acid content of the urine can be calculated.

G. W. CAMBRIDGE

**768. Circular-paper chromatography. V. Separation, identification and quantitative determination of urea with application to human urine.** Tara Rao and K. V. Giri (*J. Indian Inst. Sci.*, A, 1953, **35** [2], 137-143).—Classical methods of urea determination, which are reviewed, are unsuitable for the determination of urea in biological fluids owing to the

presence of other constituents. Circular-paper chromatography is used to separate the urea in urine; test samples are chromatographed and the position of the urea is determined by spraying half of the paper with phenol-hypochlorite reagent. The urea band on the unsprayed half of the paper is cut out and eluted with water; the eluate is added to a soln. of  $\alpha$ -isonitrosopropiophenone reagent and heated at 100° C for 1 hr., the colour intensity of the soln. being determined colorimetrically. The method is more rapid than the usual methods of analysis and can be applied to other compounds also present in blood and urine.

H. SUTCLIFFE

**769. A new method for the determination of thiamine in urine.** J. Bouman (*Int. Rev. Vitamin Res.*, 1953, **25**, 83).—The determination of thiamine in urine is vitiated by the formation of a substance,  $\text{F}_2$ , formed from N-methylnicotinamide in alkaline soln. This is extracted by isobutanol and fluoresces. In the method described the urine sample is oxidised in acid soln. with potassium ferricyanide, the excess of ferricyanide is removed with ascorbic acid, and interfering substances are extracted by isobutanol. Thiamine left in the aq. phase is oxidised to thiochrome by alkaline ferricyanide in the usual manner, the thiochrome is extracted with isobutanol and the fluorescence is measured.

D. C. M. ADAMSON

**770. Determination of coproporphyrin for clinical purposes.** J. Brugsch and F. Kubowitz (*Biochem. Z.*, 1953, **324** [3], 244-248).—A simple procedure for the determination of coproporphyrin (I) in urine by comparison of the red fluorescence of the extracts in u.v. light with that of standard solutions of the coproporphyrin-I tetramethylester is described. The extracts must be diluted with 5 per cent. HCl until they contain from 0.05 to 0.1  $\mu\text{g}$  of coproporphyrin per ml, when the intensities of fluorescence are proportional to the concentrations. As a check on the method and for identification purposes the u.v. spectrum is plotted; molar absorption coefficients,  $\epsilon$ , for coproporphyrin-I tetramethylester in 5 per cent. HCl and in dioxan solutions are reported for the range from  $\lambda = 700 \text{ m}\mu$  to  $\lambda = 260 \text{ m}\mu$ . The value found by Strait *et al.* (*J. Appl. Physiol.*, 1952, **4**, 699) for the main band max. of I in 5 per cent. HCl at  $\lambda = 402 \text{ m}\mu$ , viz.,  $\epsilon_{\text{cm}}^{1\%} = 6800$  and which affords  $\epsilon_{\text{cm}} = 102 \times 10^3 [\text{cm}^2/\text{Mol I}]$ , is confirmed.

W. MANZ

**771. Paper-chromatographic analysis of bile pigments.** W. Stich, R. Kehl and H. R. Walter (*Hoppe-Seyl. Z.*, 1953, **292**, 178-180).—Paper chromatographic analysis of bile pigments can be carried out in 2:4-dimethylpyridine-water, methanol-water or acetone-water, at certain concentrations and with addition of aq.  $\text{NH}_3$ .  $R_F$  values have been measured for bilirubin, mesobilirubin, urobilin, stercobilin and neoxanthobilirubin acid. The method represents an advance in method of differentiation of bile pigments.

P. HAAS

**772. The fractionation of urinary neutral 17-ketosteroids by adsorption and partition chromatography.** E. E. Cook, S. R. Stith, A. E. Hall and M. P. Feldman (*Analyst*, 1954, **79**, 24-29).—The fractionation of urinary neutral 17-ketosteroids by adsorption chromatography by the method of Pond (*Brit. Abstr. C*, 1952, 158) and by partition chromatography by the method of Jones *et al.* (*Brit. Abstr. C*, 1953,



265) is described. The steroids in the eluates after removal of solvent are determined absorptiometrically by the Zimmerman reaction; the identities of those occurring in each fraction are ascertained by comparison with pilot substances. Both methods show the constancy of the chromatographic patterns for urines from some subjects over a period of weeks. A. O. JONES

**773. The determination of formaldehydogenic steroids.** D. C. Smith and S. L. Tompsett (*Analyst*, 1954, **79**, 53-54).—The neutral steroid extract obtained after hydrolysis of urine with hot dil. mineral acid contains material that reacts with  $\text{HIO}_4$  to liberate formaldehyde (Tompsett, *J. Clin. Path.*, 1953, **61**, 74), which may be determined by the chromotropic acid reaction (Tompsett *et al.*, *Brit. Abstr. C*, 1953, 384). Deoxycorticosterone and corticosterone when added to urine could thus be recovered quantitatively. Experiments were made to determine possible interference by fatty residues that may occur in urinary extracts. With triolein and lecithin the first three 3-ml fractions distilled from a 15-ml reaction mixture contained only small amounts of formaldehyde; subsequent fractions contained more. Fractionation of urinary extracts showed that these extracts do contain substances related to triolein or lecithin, but their effect can be minimised by collecting only the first 9 ml of distillate. With vitamin-A acetate 50 per cent. of the theoretical formaldehyde was found. Carotene behaved similarly to triolein. When either of these was added to urine no additional formaldehyde was detected in the extracts, the power to produce it being destroyed in the hydrolytic treatment. A. O. JONES

**774. Micro-estimation of formaldehydogenic steroids by bismuthate oxidation.** R. W. H. Edwards and A. E. Kellie (*Biochem. J.*, 1954, **56** [2], 207-211).—A method for the micro-determination of formaldehydogenic steroids by means of  $\text{NaBiO}_3$  as the oxidant is described. The isothermal distillation and subsequent colorimetric determination of the formaldehyde takes place at  $100^\circ\text{C}$  in a stoppered Conway unit. The determination is complete within 3 hr., and the method is suitable for batch determinations. The method has an accuracy of  $\pm 1.5$  per cent. in the range 5 to 25 millimicromoles. B. VINEY

**775. Estimation of pregnanediol in human urine.** II. T. Dati, G. de Angelis and C. Luly (*Ric. Sci.*, 1953, **23** [11], 1979-1987).—Examination of the absorption spectra shows that the purification of pregnanediol from human urine cannot be accomplished by chromatography on an  $\text{Al}_2\text{O}_3$  column and by elution with acetone or ether. Separation can be effected by ascending paper chromatography and elution is satisfactory with a mixture of benzene and xylene saturated with 2 N HCl. S. K. LACHOWICZ

**776. New method for estimation of oestrogens.** D. Pontius (*Klin. Wschr.*, 1953, **31**, 1010).—The dried residue from urine prepared by the Stevenson-Marrian method (*Brit. Abstr. C*, 1948, 13) is dissolved in chloroform, washed with bicarbonate and dried with  $\text{Na}_2\text{SO}_4$ . The  $\text{CHCl}_3$  soln. is divided into 2 portions and each is evaporated. To one residue are added 3 drops of  $\text{HClO}_4$  and 1 drop of  $\text{CHCl}_3$  saturated with picric acid. The mixture is placed in a bath of boiling water for 5 min. After cooling, one tube (the blank) is treated with 2 ml of peroxide-containing ether (ether shaken with Perhydrol)

and 2 ml of a mixture of phenol and  $\text{CHCl}_3$  (2 + 5). The other tube is treated with phenol-chloroform mixture and peroxide-free dry ether. The extinctions are measured in an Elko photometer and standard calibration curves are prepared with the aid of pure oestrogens. G. W. CAMBRIDGE

**777. The determination of amino-acids in the presence of ammonia and uric acid.** Z. M. Zielińska (*Acta Biol. Exp., Lodz*, 1952, **16**, 265-270).—The method of Folin for the determination of the  $\alpha$ -amino N of amino-acids was used. The presence of small quantities of uric acid did not affect the estimation; quantities greater than  $10\text{ }\mu\text{g}$  in a sample containing 15 to  $30\text{ }\mu\text{g}$  of amino-acid caused considerable increases in the value for amino-acid N. Ammonia N in quantities not exceeding a few  $\mu\text{g}$  is determined as amino-acid N. D. F. HAWKINS

**778. Circular-paper chromatography. VI. The quantitative determination of amino-acids.** K. V. Giri, A. N. Radhakrishnan and C. S. Vaidyanathan (*J. Indian Inst. Sci., A*, 1953, **35** [2], 145-180).—The following methods of amino-acid analysis are reviewed: (i) isotope dilution, (ii) enzymatic, (iii) microbiological, (iv) chromatographic, including starch-column chromatography, the use of ion-exchange resins and paper chromatographic methods. Methods for the quantitative analysis of amino-acids after chromatography on paper are placed in the following categories: (i) visual comparison of the intensity of the colour produced with ninhydrin with that of a standard sample, and other physical methods; (ii) isotope-dilution analysis of the spots; (iii) elution of the amino-acid band and estimation with ninhydrin or copper phosphate; (iv) elution of the ninhydrin stained band; (v) retention analysis; and (vi) serial dilution method.

The greater part of the article deals with the use of circular-paper chromatography. Variations in metal ions, concn. of ninhydrin, ninhydrin solvent, temp. for colour development and time kept at the colour developing temp., and the relation between the area of the band, colour intensity,  $R_F$  values and distance travelled by the solvent front are found to effect the resultant chromatogram and must be controlled. After development, the chromatogram is air-dried, stained, dried again and eluted, and the amino-acid is estimated colorimetrically, calibration curves being previously prepared for each amino-acid under identical conditions. The method can be satisfactorily applied to the hydrolysate of edestin. H. SUTCLIFFE

**779. Circular-paper chromatography. VII. A technique for the separation and estimation of "overlapping amino-acids."** K. V. Giri and N. A. N. Rao (*J. Indian Inst. Sci., A*, 1953, **35** [4], 343-353).—A technique is described for separating amino-acids that overlap when developed with a solvent mixture of *n*-butanol, acetic acid and water. Circular paper chromatography is used with solvent mixtures such as pyridine, pentyl acetate and water. By preparation of standard mixtures for comparison, quantitative determinations have been made. A. WEBSTER

**780. Electrophotometry of amino-acids on paper chromatograms.** A. A. Christomanos (*Enzymologia*, 1953, **16**, 87-90).—By measurement of the extent and depth of ninhydrin colour of amino-acid spots on a paper chromatogram, the constituents of an amino-acid mixture can be roughly estimated. J. J. SCOTT

**781. Electrochromatography [of amino-acids] on paper.** D. P. Burma (*Anal. Chim. Acta*, 1953, **9** [6], 518-524).—The term "electrochromatography" is used to denote the simultaneous operation of chromatography and ionophoresis. The electric field can be applied either in the direction of flow of the solvent or at right angles to it. The filter-paper is previously treated with a buffer solution and the developing solvent is saturated with it. Colloidal graphite painted on the edges of the paper serves as electrodes. A study has been made of a number of amino-acids at various pH values with a mixture of phenol and a buffer soln. as the developing solvent. On ordinary paper, acidic amino-acids move towards the anode and basic ones towards the cathode, but neutral amino-acids do not move at all. With buffer-treated papers the movement depends upon the pH of the buffer and the iso-electric points of the amino-acids. By choice of suitable pH and application of electric field in proper direction amino-acids having very close  $R_F$  values (except isomers) can be separated from one another. W. C. JOHNSON

**782. "Multiple dipping" procedures in paper chromatography: [and] a specific test for hydroxyproline.** J. B. Jepson and Ivor Smith (*Nature*, 1953, **172**, 1100-1101).—A "multiple dipping" procedure is described in which paper chromatograms are dipped successively in some or all of the reagents ninhydrin, isatin, Ehrlich reagents, a Sakaguchi reagent (application of 0.1 per cent. 8-hydroxyquinoline in acetone, air drying followed by dipping into 0.2 ml of bromine in 100 ml of 0.5 N NaOH soln.) and a diazo reagent (from amyl nitrite and *p*-anisidine in ethanol). Between each application of reagent the chromatogram is left to develop fully. This multiple dipping technique is useful for confirming, by specific reagents, the nature and position of components first located by a general reagent, for revealing the composite nature of a single spot, and for revealing two or more sets of components that do not each respond to the same general reagent. In general, the usual colour responses and sensitivities of amino-acids to the reagents are almost unaltered by previous treatment. An important exception gives a highly sensitive and specific test for hydroxyproline. After heating with water, the duck-egg blue colour due to hydroxyproline is replaced by an intense purplish-red (cerise) colour within a few seconds of applying Ehrlich reagent over it. The effect is not seen when either acid or *p*-dimethylaminobenzaldehyde is applied alone. This colour test will detect hydroxyproline on paper chromatograms at a concn. of 0.1  $\mu\text{g}$  per sq. cm, and is specific for hydroxyproline among more than 120 amino-acids, amines and heterocyclic acids tested. I. JONES

**783. Quantitative evaluation of the paper chromatography of protein hydrolysates.** F. G. Fischer and H. Dörfel (*Biochem. Z.*, 1953, **324** [7], 544-566).—A method is described for the estimation of the amino-acids in 5 to 10 mg of protein. Unidimensional chromatography of the protein hydrolysate is carried out by the method of McFarren and Mills (*Brit. Abstr. C*, 1952, 347). After development with ninhydrin, the amino-acids in the spots were converted to the Cu salts for estimation. Control amino-acid mixtures were run with each investigation. Details are given of each stage of the separation and estimation, and the limits of accuracy are clearly defined. G. W. CAMBRIDGE

**784. Routine colorimetric method for estimation of small quantities of protein in body fluids.** E. Böhle and Herbert Fischer (*Klin. Wschr.*, 1953, **31**, 798-802).—The method is based on the combination of protein with an acidic dye and subsequent estimation of the lowering of the concn. of the dye in the supernatant fluid. The estimation is carried out in small centrifuge tubes graduated at 1 ml and 3 ml. The reagent is 0.001 M Amido Black soln. in citric acid buffer (pH 2.2). One ml of the protein soln. is mixed with 2 ml of the reagent and the mixture is set aside at room temp. for 15 min. and then centrifuged for 10 min. at 3000 r.p.m. One ml of the supernatant liquid is then diluted to 25 ml with distilled water. The colour is estimated in a 1-ml cuvette in a photometer with a 578 filter. For total protein estimations undiluted soln. is used. For globulin estimation, 1 ml of the soln. is treated with 1 ml of saturated  $(\text{NH}_4)_2\text{SO}_4$  soln. and centrifuged after 2 hr. The ppt. is washed with half-saturated  $(\text{NH}_4)_2\text{SO}_4$  soln. and then dissolved in 1 ml of 0.9 per cent. aq. NaCl soln.; the estimation is carried out as before. As albumin, globulin, protamine and histone take up different amounts of dye they can be differentiated. The estimation gives an indication of the number of basic groups in the mol. In body fluids such as serum, puncture fluids and cerebrospinal fluid only proteins take up the dye, but in urine higher peptides are also estimated. Amino-acids, creatine, uric acid and urea are without effect on the estimation. Twenty  $\mu\text{l}$  of human serum are required for albumin and globulin estimation and the values obtained agree closely with those obtained by the Antweiler method. Two ml of cerebrospinal fluid are required for albumin and globulin estimation and the values obtained are significantly higher than those found by the Kafka method. However, estimation of total N gives values in good agreement with those by the dye method. The estimation of protein in urine requires 4 ml of fluid and tests have indicated a daily excretion of 10 to 20 mg (higher peptides included). The method can also be applied to de-natured proteins which show a diminished uptake of dye. G. W. CAMBRIDGE

**785. Estimation of water content of small amounts of proteinaceous material.** H. Sobel (*Anal. Chem.*, 1953, **25** [11], 1756).—The method is used for the determination of water in 5- to 50-mg specimens of lyophilised material. Dilute Karl Fischer reagent (prepared by adding the conc. reagent to anhydrous methanol until a yellow colour develops) is used. The reagent is added to the lyophilised material, the mixture is centrifuged and the water in the supernatant liquid is determined colorimetrically. G. P. COOK

**786. Estimation of D- and L-glutamic acid in proteins.** G. H. Wiltshire (*Biochem. J.*, 1953, **55** [1], 46-49).—The L- and total glutamic acid has been estimated in acid hydrolysates of 3 purified proteins and certain protein preparations. Total glutamic acid was estimated by determination of amino nitrogen after its separation on ion-exchange columns (Amberlite IR-4B), the purity of the glutamic acid being checked by paper chromatography. L-Glutamic acid was estimated by means of its enzymic decarboxylation with washed cell suspensions of *Clostridium welchii* S.R.12. D-Glutamic acid was then estimated by difference. The racemisation of L-glutamic acid, under the conditions of acid hydrolysis and subsequent treatment of the test hydrolysate, has been followed

polarimetrically, and it is shown that under such conditions 5 per cent. of the D-isomer was produced from L-glutamic acid. The combined results indicate that D-glutamic acid in excess of that estimated to be formed by inversion is present in hydrolysates of two proteins that had been treated with dil. alkali and in the cells of *Lactobacillus casei*, but not in three purified animal or plant proteins or in tobacco-mosaic-virus protein. Results by the decarboxylase method are accurate to  $\pm 5$  per cent. and those by the Amberlite-resin method to  $\pm 2$  per cent.

P. CHAPLEN

**787. The colorimetric determination of 3:5-di-iodotyrosine.** N. R. Moudgal, L. K. Ramachandran and P. S. Sarma (*Analyst*, 1954, **79**, 43-46).—When one of the iodo groups of di-iodotyrosine is replaced by a nitroso group the resulting compound can form metallic chelates. This qual. reaction has been adapted for quant. purposes, the green complex with Co being the most convenient for colorimetric measurement. The max. absorption is at 390-395 m $\mu$ . For 0.1 to 0.9 mg in a final vol. of 10-45 ml the relation between optical density and concn. of di-iodotyrosine is linear. A procedure is given for hydrolysis of natural and artificial iodoproteins and separation of thyroxine from di-iodotyrosine by butanol fractionation. The determination of di-iodotyrosine in 500 mg of a natural or artificial iodoprotein can be carried out with an accuracy of  $\pm 2.8$  per cent.

A. O. JONES

**788. The fluorimetric determination of polycarboxylic acids following chromatography.** C. E. Frohmann and J. M. Orten (*J. Biol. Chem.*, 1953, **205** [2], 717-723).—A method is presented for the fluorimetric determination of polycarboxylic acids after their separation by chromatography on silica gel. Recoveries from mixtures of the pure acids and from tissues show that the fluorimetric method is at least as accurate as the more laborious titrimetric method previously used. The derivatives formed during the determinations of the acids can be extracted with ether for subsequent measurement of radioactivity. The levels and radioactivity of the acids of the tricarboxylic acid cycle were measured in the livers of rats previously injected with acetate-1-<sup>14</sup>C. The results suggest the possibility of a source of succinate other than acetate.

B. VINEY

**789. Turbidimetric estimation of hyaluronate.** R. H. Pearce (*Biochem. J.*, 1953, **55** [3], 472-477).—The effects of pH and of the concn. of hyaluronate, protein and electrolyte on the turbidity-producing reaction between hyaluronate and protein and the kinetics of the reaction have been studied. The turbidity produced is a simple power function of the concn. of hyaluronate and depends on the properties of the particular specimen (principally its purity and protein content). The optimum pH is 4.1 to 4.2. Electrolytes interfere with the formation of turbidity, NaCl being more active in this respect than acetate. The increased sensitivity achieved by raising the albumin concn. is offset by a loss in precision. The turbidity develops rapidly at a rate dependent on the law of mass action, and it decays at a diminishing rate over a period of several hours. The application of these observations to some practical analytical problems is illustrated.

B. VINEY

### Drugs

**790. Separation of some of the opium alkaloids by surface chromatography.** M. L. Borke and E. R.

Kirch (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [10], 627-629).—A buffered fluorescent adsorbent (pH 6.6) and 1:4-dioxan as the solvent are used to separate the major opium alkaloids and meconic acid. The separated compounds gave violet or blue spots on a light-green background. As little as 15 to 20  $\mu$ g of papaverine hydrochloride, narcotine or meconic acid, and 50  $\mu$ g of morphine, codeine or narceine could be detected by this method.

J. CALEY

**791. The colorimetric estimation of morphine in camphorated tincture of opium.** G. Tunstall and M. P. Taylor (*J. Pharm. Pharmacol.*, 1953, **5** [10], 737-742).—A substance other than morphine was found to give Radulescu's reaction and to interfere with the colorimetric assay of camphorated tincture of opium. In samples containing tincture of opium as the source of morphine, the test showed the content to be about 10 per cent. higher than the theoretical estimate. The interfering substance can be extracted with benzene. The official method of assay of camphorated tincture of opium is improved by inserting this extraction after the addition of  $(\text{NH}_4)_2\text{SO}_4$ .

E. H. JOHNSON

**792. Detection of minor alkaloid constituents in quinine. Scheme for separation by paper chromatography.** P. Castille (*Pharm. Weekbl.*, 1954, **89** [1-2], 1-11).—In order to minimise the masking effect of the quinine trail on the spots formed by the minor constituent alkaloids, 1.25 N aq. potassium oxalate (in which quinine is slightly sol.) is used as ascending solvent on paper that has previously been soaked in a 0.02 M soln. of the same salt and then dried. A  $\text{CHCl}_3$  soln. of the alkaloids is prepared by shaking an alkaline soln. of the sample ( $\approx 50$  mg of total alkaloids) in 5 ml of water with  $\text{CHCl}_3$  (5 ml). Three spots containing, respectively, 10, 20 or 40  $\mu$ g of alkaloids, alternating with 3 spots containing, respectively, equal amounts of pure quinine, are started from the base-line; development (30 to 45 min.) is carried out in a covered 1-litre beaker, the atmosphere in which is kept moist by means of filter-paper dipping into water. After exposure of the moist chromatogram to iodine vapour, spraying with ethyl acetate slightly acidified with  $\text{H}_2\text{SO}_4$  and drying, the chromatogram is examined in u.v. light. Examination of the most suitably developed spots and comparison of these with spots developed from alkaloid mixtures of known content reveals the presence of 0.25  $\mu$ g of quinidine, 1  $\mu$ g of hydroquinine, 0.5  $\mu$ g of chichonine or 0.5  $\mu$ g of cinchonidine. Further detailed guidance is given for the evaluation of the chromatogram.

P. S. ARUP

**793. Some assay processes involving the use of ion-exchange resins.** L. Saunders, P. H. Elworthy and R. Fleming (*J. Pharm. Pharmacol.*, 1954, **6** [1], 32-37).—Experimental procedures and results are given for the estimation, by means of ion-exchange processes, of quinine in ethanolic soln. and quinine in quinine salts and of glycine in the presence of gelatin and for the direct estimation of ephedrine hydrochloride. By the methods described results with an error as small as  $\pm 0.5$  per cent. can be obtained.

N. M. WALLER

**794. Digitoxin.** P. Demoen and P. Janssen (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [10], 635-640).—The possibilities and limitations of some chromatographic and spectrophotometric techniques used in the analysis of digitoxin and related

substances are discussed. All the samples tested were found to contain impurities; the characters recorded in the literature are not usually applicable to chemically pure digitoxin. J. CALEY

**795. The spectrophotometric determination of phenylacetone in amphetamine.** S. K. Freeman (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [10], 621-622).—Alcoholic solutions of carefully purified phenylacetone show fine structure in the region of 260  $m\mu$  and a broad spectrum band in the neighbourhood of 285  $m\mu$ . In order to determine the amount of phenylacetone in amphetamine, solutions containing up to 10 per cent. of the ketone are measured at 290  $m\mu$ ; the amount is calculated from a standard graph. The standard deviation is  $\approx \pm 0.15$  per cent. J. CALEY

**796. Coulometric determination of small quantities of sulphanilamides.** K. Sykut (*Ann. Univ. M. Curie-Skłodowska, AA*, 1951, **6**, 47-61).—Determination of sulphanilamides by coulometric bromination with a platinum-silicon carbide electrode pair is described. Accurate measurement of the current flowing through the cell and of the time of bromination permits determination of small (0.2 mg) quantities of sulphanilic acid and sulphanilamides with an error of about  $\pm 0.2$  per cent. S. K. LACHOWICZ

**797. A colorimetric method for the determination of small amounts of procaine present as procaine benzylpenicillin B.P. in veterinary food products.** M. G. Ashley and J. F. Lees (*J. Pharm. Pharmacol.*, 1954, **6** [1], 50-54).—A colorimetric method is described for the determination of between 3.0 per cent. and 2.5 p.p.m. of procaine benzylpenicillin in animal foodstuffs, premixes and concentrates. The food sample is extracted with  $\text{CHCl}_3$  from alkaline soln. An aliquot of this extract is extracted with 0.1 *N* HCl, and an aliquot of this soln. is diazotised and coupled with *N*-(1-naphthyl)ethylenediamine. The optical density of the test soln. is measured and the procaine benzylpenicillin content is determined from a calibration graph. N. M. WALLER

**798. A colorimetric method, based on metallic complex formation, for the detection of aureomycin in presence of amino-acids and proteins.** A. E. Oxford (*Nature*, 1953, **172**, 395-396).—Aureomycin forms stable yellow complexes sol. in *n*-butanol with certain divalent cations, notably Ca and Co in alkaline soln. These can be demonstrated in the presence of other divalent cations by taking aureomycin (200 to 400  $\mu\text{g}$  per ml),  $\text{Ca}^{++}$  or  $\text{Co}^{++}$  (20 to 40  $\mu\text{g}$  per ml), "Bactogelatin" (1000  $\mu\text{g}$  per ml) (Ca free) and aq.  $\text{NH}_3$  to 0.25 *M* or NaOH to 0.04 *M*. This mixture is kept at 30° C for 10 min., and then extracted with *n*-butanol. Ca and Co give a yellow colour in both layers; the colour is present in the butanol layer only with Ca. Aureomycin (20  $\mu\text{g}$ ) can be detected in a soln. containing "Bactogelatin" and "artificial" casein hydrolysate by adding a few drops of  $\text{CaCl}_2$  and NaOH. A yellow colour after incubation at 30° for 15 min. indicates aureomycin. E. H. JOHNSON

**799. Soluble aspirin tablets N.F.** G. Raine (*Pharm. J.*, 1954, **172**, 31-32).—Batches of soluble aspirin tablets were submitted to tests of (i) colour and odour, (ii) solubility in  $\text{H}_2\text{O}$ , (iii) taste and pH in soln., (iv) lower limit of soluble aspirin by  $\text{CHCl}_3$  extraction after acidification and (v) limit of free salicylic acid by extraction of powdered tablet with  $\text{CHCl}_3$ , evaporation and comparison of the colour

developed by the residue with  $\text{Fe}^{+++}$  against a standard. Of 15 samples only 2 passed all tests (reasonable standards being set) and only 5 were considered suitable for use. An assay procedure is suggested which includes extraction of the powdered tablet with  $\text{CHCl}_3$ , evaporation and hydrolysis (0.5 *N* NaOH, 10 min.), dilution, acidification and adjustment to volume followed by estimation of salicylic acid in an aliquot portion by 0.5 *N* Br soln. In all  $\text{CHCl}_3$  extractions of aspirin, decomposition and loss of acetic acid are avoided by evaporating the extract partly by heat and partly at room temp.; the residue is dried in a desiccator. F. R. MUMFORD

**800. The evaluation of buffer antacids, with particular reference to preparations of aluminium.** D. N. Gore, B. K. Martin and M. P. Taylor (*J. Pharm. Pharmacol.*, 1953, **5** [10], 686-691).—The acid-neutralising capacity test in B.P.C. 1949 and in U.S.P. XIV, is inadequate for testing the therapeutic value of dried  $\text{Al}(\text{OH})_3$  gel. In the test suggested, 1.0 g of sample is added to 200 ml of water and 3 ml of *N* HCl, with constant agitation. The pH is determined electrometrically at 5-min. intervals for 30 min.; then 1 ml of HCl is added every 10 min. and the pH is determined before each addition, the test being continued until the target pH set for the particular agent is reached. The test determines rate of effect, pH value, and the capacity to maintain the effect while fresh gastric juice (*in vivo*) is secreted. The results of the test with Al glycinate, and different samples of dried  $\text{Al}(\text{OH})_3$  gel are given. E. H. JOHNSON

**801. The chemical evaluation of antacids.** H. Brindle (*J. Pharm. Pharmacol.*, 1953, **5** [10], 692-708).—A test for antacids, which could be standardised and which shows the probable rate of action and also the ultimate pH that will be reached in the stomach, is described. The artificial gastric juice used is 0.05 *N* HCl with 0.15 per cent. each of pepsin, peptone and NaCl. The average pH at 38° C is 1.5. The "acid neutralisation value" of an antacid (the amount that neutralises 100 ml of 0.05 *N* HCl) is found by subjecting the antacid to excess of acid at 38° C for 4 hr. The rate of neutralisation and the final pH can be determined by using a 20 per cent. excess of antacid and noting the pH frequently. The results with a number of antacids in common use are given. Bismuth carbonate was inactive. E. H. JOHNSON

**802. Methods of testing essential oils.** British Standards Institution (B.S. 2073 : 1953, 23 pp.).—Physical methods are described for sp. gr., apparent density (wt. per ml), optical rotation, refractive index, freezing-point and melting point, and solubility in alcohol. The chemical methods include acid val. and ester val., ester val. after acetylation and the determination of citronellal, aldehydes other than citronellal, carvone and menthone, and phenols. The analytical methods recommended are well-tried and modified in some details for greater accuracy. The conditions for the determination of solubility in alcohol are more strictly defined and a "standard opalescence" prepared from  $\text{AgNO}_3$  and NaCl is used for comparison when the soln. is turbid. N. E.

**803. A note on the assay of ascaridole in castor oil solution.** D. C. Garratt and R. F. Phippen (*J. Pharm. Pharmacol.*, 1954, **6** [1], 60-61).—Oil of chenopodium in castor oil solution (50 g) is distilled with ethylene glycol (50 ml) at reduced pressure



The distillate is diluted with water (100 ml) and extracted four times with ether (25 ml), the extract being shaken with 90 per cent. acetic acid (40 ml) before evaporation, to avoid loss of ascaridole. After evaporation the remaining soln. is adjusted to 50 ml with 90 per cent. acetic acid and the B.P. method is then used. Results have been satisfactory.

N. M. WALLER

**804. Anaesthesia. XLIII. The estimation of anaesthetic trifluoroethyl vinyl ether in aqueous solution.** F. K. Bell and J. C. Krantz, jun. (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [10], 633-634).—An empirical procedure based on the observation that under certain conditions a bromine soln. is decolorised by an aqueous soln. of trifluoroethyl vinyl ether; the degree of decolorisation is proportional to the amount of the ether. This method is simple and can be completed in 40 min. and would seem to be applicable to the examination of numerous other substances reactive in a similar manner towards bromine water.

J. CALEY

**805. A bio-assay method for the evaluation of adrenocortical potency by means of experimental trauma, with shock, in rats.** V. Schenker (*Endocrinology*, 1953, **53** [4], 345-357).—A method of bio-assay of the "stress survival" type for the evaluation of adrenocortical activity in biological materials and in crystalline compositions is described. Reproducible trauma with shock is experimentally induced by a modified form of the "Noble-Collip Drum." The response is expressed as percentage survival following cessation of drum stress in fasting pre-treated adrenalectomised male albino rats. The standard of reference was "Compound F," which is active at a dosage level of 15  $\mu$ . The method has been applied to cortisone, corticosterone and an adrenocortical extract.

N. E.

**806. Bacterial survival in systems of low moisture content. V. Comparison of tests for sterility of oils.** K. Bullock and N. H. Booth (*J. Pharm. Pharmacol.*, 1953, **5** [10], 757-771).—Oil containing as few as 4 spores in 10 g is prepared by addition of heavily contaminated peptone to the oil and then dilution with sterile oil. A limiting viable-count technique has been elaborated. Five grams of oil are mixed with 5 per cent. of sterile milled peptone and 5 ml of light petroleum are used as solvent. The solvent is removed after centrifuging and the deposit is taken up in water (10 ml for 5 g of oil sampled) and a quintuplicate roll-tube count on 1 ml of aq. suspension is made. If all 5 tests are negative the oil is sterile. This test is found to be more sensitive than the B.P. test for sterility or the filtration test of Davies and Fishburn.

E. H. JOHNSON

See also Abstract 733.

## Food

**807. Synthetic colouring matters in foodstuffs.** A. Castille (*An. Bromatologia*, 1953, **5** [3], 265-275).—The isolation and chromatographic separation of dyes from foodstuffs is described. The methods of extraction applied to different kinds of food are indicated and the results of chromatographic separation by conventional methods are given.

H. PRITCHARD

**808. Paper chromatography of synthetic colouring matters in foodstuffs. I. Water-soluble yellow and orange dyestuffs.** A. Charro (*An. Bromatologia*,

1953, **5** [3], 359-373).—Paper chromatography has been applied to the separation of dyestuffs from foodstuffs, the disc method of Rutter (*Brit. Abstr. C*, 1950, 215) being selected. The behaviour of the permitted yellow and orange colours is recorded and tabulated in terms of  $R_F$  values, and a comparison is made with 42 saffrons and similar condiments on the Spanish market. It was possible by this means to detect added colour in all but 7 of them.

H. PRITCHARD

**809. Determination by paper chromatography of glycols in foodstuffs.** K. E. Bergner and H. Sperlich (*An. Bromatologia*, 1953, **5** [3], 241-244).—The degree of toxicity of glycols and similar derivatives used as solvents and humectants in foods, tobacco and cosmetics varies with the type. Their effects range from that of ethylene glycol to that of butane-1:3-diol, which is little more harmful than glycerol. Similarity of chemical composition renders normal methods of separation difficult, but paper chromatography is effective. The glycols were extracted with ether and the bands were made visible after chromatography with a solution of 9 pt. of 5 per cent.  $\text{AgNO}_3$  and 1 pt. of 25 per cent.  $\text{NH}_3$ . Most of the glycols can be separated with ethanol-chloroform mixtures; to distinguish butanediol from butyleneglycol, ether saturated with water is recommended.

H. PRITCHARD

**810. Nitrogenous fraction in milk from cows, goats and sheep.** O. A. Valenciano (*An. Bromatologia*, 1953, **5** [3], 255-263).—A comparison has been made of the food value of the milk of cows, goats and sheep.

Representative samples of each type of milk were examined for total nitrogen ( $\text{N} \times 6.38$ ) by the Kjeldahl method and other nitrogenous fractions were determined as follows: casein ( $\text{N} \times 6.38$ ) was determined by pptn. with 1 per cent. acetic acid and filtration, N being determined by the Kjeldahl method on the filtrate. A further portion of the filtrate was neutralised with 10 per cent. NaOH and divided into two equal parts. To one part was added 0.3 ml of 10 per cent. acetic acid, the mixture was boiled to precipitate lactalbumin ( $\text{N} \times 6.38$ ), filtered and N was determined by the Kjeldahl method. The second part was saturated with  $\text{MgSO}_4$  to precipitate lactoglobulin ( $\text{N} \times 6.38$ ). A 100-ml portion of the rest of the original filtrate was treated with 10 per cent. sulphuric acid and 10 per cent. phosphotungstic acid soln. Nitrogen was determined on the filtrate,  $\text{N} \times 9.35$  being used to express non-protein nitrogen. The average figures found were:

|                           | Cow  | Goat | Sheep |
|---------------------------|------|------|-------|
| Casein, per cent. ..      | 2.30 | 2.39 | 3.98  |
| Albumin, per cent. ..     | 0.33 | 0.31 | 0.41  |
| Globulin, per cent. ..    | 0.11 | 0.09 | 0.102 |
| Non-protein, per cent. .. | 0.24 | 0.29 | 0.29  |

H. PRITCHARD

**811. A method for determination of lactose in milk by paper chromatography.** C. J. Honer and S. L. Tuckey (*J. Dairy Sci.*, 1953, **36**, 1233).—A quantitative method for determining lactose in milk is described consisting in (i) paper chromatography, (ii) reaction with alkaline ferricyanide and (iii) reaction of the resulting ferrocyanide with ferric ions and measurement of the colour of the ferric-ferrocyanide complex. The method is specific for lactose.

**Procedure**—Dilute 10 g of milk to 100 ml with water and place 0.01 ml 5 in. from one end of each of two strips of Whatman No. 1 filter-paper, about

22½ in. long. Dry the strips quickly in a stream of warm air, fold at a line 4 in. from the end and immerse the short end in a trough of the developing solvent (the non-aqueous layer of a vigorously-shaken mixture of 2.5 pt. ethyl acetate, 1 pt. pyridine and 3.5 pt. distilled water). After setting aside in a closed insulated container for 24 hr., immerse one strip in aq.  $\text{AgNO}_3$ -acetone soln. (5 ml saturated  $\text{AgNO}_3$  and 95 ml acetone) in order to locate the lactose spot, and use this as a guide when cutting the other strip. Dry the second strip for 6 hr. at room temp. *in vacuo*, and cut the strip in the region of the lactose spot into 10 segments about 0.1 in. wide. Place each in a 4-ml calibrated test tube and to each tube add 0.5 ml  $\text{K}_3\text{Fe}(\text{CN})_6$  (0.1 per cent. in water) and 0.5 ml  $\text{Na}_2\text{CO}_3$ -KCN soln. (0.8 g  $\text{Na}_2\text{CO}_3$  and 0.25 g KCN in 100 ml of water). Place in boiling water for exactly five minutes, cool to below 30°C in a bath maintained at 20° to 25°C and, after 10 min., add  $\text{FeSO}_4$ -gum arabic soln. (1.5 g anhyd.  $\text{FeSO}_4$ , 1 g gum arabic U.S.P. in 1 litre of distilled water containing 1 ml conc. sulphuric acid). Mix thoroughly by inversion and after 15 min. read the extinction at 600 m $\mu$  in a spectrophotometer. A blank is similarly prepared with Whatman No. 1 filter-paper as described. The readings for the lactose-containing segments start at a minimum increase to a peak and then decrease to a minimum. The total of the readings is related to the lactose concentration, which is read off from a graph prepared by chromatographing 5, 10, 20, 30, 40 and 50  $\mu\text{g}$  of lactose and proceeding as above. An optical density of 1.0 was found for 20  $\mu\text{g}$  of anhydrous lactose.

For a sample of milk, the mean value found was 4.516 per cent. anhyd. lactose, standard deviation  $\pm 0.16$  per cent. The recovery of added lactose varied between 92 and 97 per cent. and averaged 95 per cent.

D. C. M. ADAMSON

**812. Separation of sodium, potassium, magnesium and calcium in milk ash by ion-exchange chromatography.** W. J. L. Sutton and E. F. Almy (*J. Dairy Sci.*, 1953, **36**, 1248).—A simple method is described for the separation of Na, K, Mg and Ca in salt mixtures and milk ash by ion-exchange chromatography. A column of Dowex-50 61 cm long and 23 mm in diameter was flushed with 0.7 N hydrochloric acid for 3 to 4 hr. at a flow rate of 2.5 ml per minute; a 10-ml portion of the sample soln. in 0.7 N HCl was then added to the top of the column, and was allowed to drain slowly down until level with the top of the column. Then 0.7 N HCl was added to give a layer a few cm deep above the column. Input of acid and outflow were adjusted to maintain this level at a flow rate of 2.3 ml per min. The first 300 ml of eluate were free from cations, and the sodium was completely eluted at 500 ml and the potassium at 700 ml. These volumes should be determined for each batch of resin. Elution with N HCl was then started and all the Mg was found in the 900 to 1150-ml fraction; the calcium followed between 1200 and 1500 ml.

These fractions were collected separately and assayed by evaporating aliquots of the well-mixed solutions to dryness to remove HCl and titrating the residue of metallic chloride with 0.02 N  $\text{AgNO}_3$  soln.

D. C. M. ADAMSON

**813. A test cell for the measurement of the conductivity of butter and margarine.** J. H. Prentice (*J. Sci. Instrum.*, 1954, **31** (1), 29-30).—The cell described is designed for use with substances of

low electrical conductivity that are prone to structural damage during sampling. The electrodes made from two cut-down cork borers of slightly different diameters can be inserted independently into the sample.

B. J. W.

**814. Methods for the sampling and chemical analysis of rennet casein.** British Standards Institution (B.S. 1416: 1953, 17 pp).—In this revised British Standard (see *Brit. Abstr. C*, 1949, 57) are described methods of sampling with a simplified sampling tube and prep. of the sample for testing. Details are given for determining particle size (sieving method), loss on drying, ash, fat (solvent extraction of HCl-digested material), iron (colorimetric), N (Kjeldahl method), wet heat resistance (replacing heat discoloration test) and for the electrometric determination of pH (replacing colorimetric method); a new test is included for determining dirt. Tests for acidity and for water-extractable lactose have been deleted and reference to ground casein is omitted.

S. C. JOLLY

**815. Relation of grade colour value of flour to rate of extraction and type of wheat.** C. R. Jones and G. J. Baker with J. A. Snodgrass and B. R. Walker (*Bull. Res. Ass. Brit. Flour-Millers*, 1954, **5** (1), 7-21).—There has been a general improvement in the colour of flour, independent of changes in milling technique as shown by tests with the Kent-Jones and Martin grading instrument on laboratory-milled flour, from wheats harvested between 1951 and 1953. The grade colour figure of laboratory-milled flour from various wheats when plotted against extraction rate gives a family of curves all approx. parallel (with one exception); hence any two flours will have a constant difference in grade colour at any extraction rate between 74 and 82 per cent. The relation between the grade colour, ash content and vitamin-B<sub>1</sub> content has been determined for flours milled from various wheats at different extraction rates. Improvements in the quality of wheat supplies are discussed and are responsible for recent improvements in the colour of national flour. Brans from different wheats have a different "specific discolouring power"—unrelated to the colour of the wheat—also a different "fragility," so that flour milled from different wheats at any given mill setting may contain different proportions of bran. A present-day grist milled to give a grade colour of 5.5 should yield a satisfactory vitamin-B<sub>1</sub> content. In one example given, a mill whose grade colour is 4.1 at 80 per cent. extraction would need an increase in extraction rate to 81.8 per cent. to reach a grade colour of 5.5. A method of calculating, from the data provided, the extraction rate required for satisfactory nutritional standards from the grade colour of the 80 per cent. extraction flour is given.

G. B. THACKRAY

**816. Determination of malic, tartaric and citric acids in fruit by ion-exchange chromatography.** H. H. Schenker and W. Rieman, III (*Anal. Chem.*, 1953, **25** (11), 1637-1639).—By the procedure described, malic, tartaric and citric acids in fruit, fruit juices or fruit products are separated by ion-exchange chromatography on Dowex-1 and then determined by oxidation with permanganate. All three acids can be determined in 8 hr., the error for each being usually about  $\pm 0.1$  mg. The sample may contain a maximum of 24 mg of any one acid. Interference by oxalic acid, in the determination of tartaric acid only, is noted.

O. M. WHITTON



817. **Saffron and its adulterants.** M. J. Fernandez Pizarro and M. Nuñez-Samper (*An. Bromatologia*, 1953, 5 [3], 349-357).—Spanish saffron, much sought after for its colouring and medicinal properties, is very expensive, consequently adulteration is widely practised. The average chemical composition of true saffron was determined and micro and macro measurements of its various structures. The adulterants may be one or more of 8 classes of substances and the means of detecting these are tabulated.

H. PRITCHARD

818. **Adulteration of pepper and its falsification with the fruits of *Schinus molle* L.** M. Nuñez-Samper (*An. Bromatologia*, 1953, 5 [3], 345-348).—Peruvian pepper, or false pepper, has appeared in many markets, particularly in Central America and also, recently, in Europe. In order to distinguish the true from the false, chemical and histological examinations were made, and the various characteristics recorded.

H. PRITCHARD

819. **Use of membrane-filter method in practical biological control in the brewery.** H. Klos and R. Reimann (*Brauwissenschaft*, 1954, [1], 10-12).—For the expeditious detection of infections, the (clear) filtered beer or rinsings from various parts of the plant are filtered (water suction pump) through a "CO<sub>2</sub>" membrane in a "COLI 5" filter (Membranfiltergesellschaft, Göttingen), after which the membrane is spread over the surface of beer-agar in a Petri dish. After > 24 hr., any beer spoilage organisms present will be clearly visible as colonies on the membrane. Membranes can be used for 5 tests apiece, and, after cleaning, are preserved in 20 to 25 per cent. ethanol. Details as to the sterilisation of the apparatus and sampling are given. Blank tests should be carried out on the water used for rinsing. Bacteriological water analyses can be expedited by the application of the above method.

P. S. ARUP

820. **Exact method for determination of fermenting capacity of yeasts of high and low capacities.** U. Hoffmann (*Brauwissenschaft*, 1954, [1], 1-6).—Comparative determinations can be satisfactorily carried out in the Warburg apparatus at 25° C, provided that the wort used in the tests and for the previous cultivation of the yeast is the same as that used in the brewery. The ratio of the fermenting capacity at 25° C to that at 7° C is almost identical for different yeasts.

P. S. ARUP

821. **Paper-chromatographic separation of thiamine and its phosphate esters.** K.-H. Kiessling and G. Lindahl (*Ark. Kemi*, 1953, 6 [3], 271-272).—Butanol saturated with water and containing 18 per cent. of formic acid is an effective solvent for the paper-chromatographic separation of thiamine and its mono-, di- and tri-phosphates in extracts of biological materials.

S. C. JOLLY

822. **Biological standardisation of essential fatty acids (a new method).** H. J. Thomasson (*Int. Rev. Vitamin Res.*, 1953, 25 [1], 62-82).—Deuel *et al.* (*J. Nutr.*, 1951, 45, 535) described a quantitative (rat) assay for essential fatty acids (Vitamin F), which required a 20-week period. By using the disturbed water metabolism of deficient animals the time is reduced to 9 weeks.

Wistar male rats are bred from parents fed on the Sherman diet (*J. Biol. Chem.*, 1924, 50, 5). At 21 days old they are fed a diet deficient in vitamin F (62 g extracted casein, 142 g sucrose, 20 g hardened coconut fat, 8.5 g salts [Jansen *et al.*, *Acta Brev.*,

1935, 5, 163], 6-8 g vitamin mixture [composition given] and 2 g extracted liver powder) and water *ad lib.* for 14 days. At the end of this period the water supply is restricted to 14 ml per animal daily and after a further 21 days the 28-day test period follows. The animals are divided into groups and individually weighed; the vitamin-free diet is fed *ad lib.* and water is restricted to 14 ml per day (apparatus described), each group in addition receiving levels between 5 and 50 mg of linoleic acid as standard or corresponding amounts of the test substance. The regression of growth response on log dose is linear and the calculation of the potency is carried out in the usual manner. The unit proposed is the activity of 10 mg of linoleic acid, on which basis results are reported for a large number of unsaturated acids and naturally occurring oils. In the latter group a highly significant correlation between spectrophotometric determinations of conjugated diene and biological activity was found.

D. C. M. ADAMSON

823. **Critical study of national and foreign methods for determining the acid value of vegetable oils and a proposed new method.** I. Vargas Fuentes (*Inf. Quim. Anal.*, 1953, 7 [6], 181-199).—Errors occur in existing methods on account of CO<sub>2</sub> dissolved in the solvent. The proposed new method requires a solvent mixture containing CS<sub>2</sub>, in which CO<sub>2</sub> has a low solubility. Ten ml of CS<sub>2</sub>, 10 ml of H<sub>2</sub>O and 10 ml of a 1 per cent. soln. of phenolphthalein in ethanol are shaken vigorously; 10 ml of the resulting emulsion are poured into the titration flask. Two to ten grams (or 2.2 to 11 ml) of oil are added and the mixture, after shaking, is titrated with 0.1 N NaOH soln. Results are  $\approx$  10 per cent. lower than those obtained by the standard method (in ethanol-ether soln.).

L. A. O'NEILL

See also Abstracts 695, 731, 732, 761, 762, 763, 797, 837.

#### Sanitation

824. **Determination of dissolved oxygen in waters in the presence of an active reducing agent.** K. Wickert and E. Ipach (*Z. anal. Chem.*, 1953, 140 [5], 350-353).—The reducing agent is oxidised by addition of bromine water, the excess of which is then removed from the examined sample of water by sulphosalicylic acid. Oxygen in water is determined by the colorimetric method (Mn<sup>++</sup> and o-tolidine), allowances being made for O introduced into the sample by reagents.

S. K. LACHOWICZ

825. **The spectrophotometric determination of ammonia in natural waters, with particular reference to sea-water.** J. P. Riley (*Anal. Chim. Acta*, 1953, 9 [6], 575-589).—In the examination of sea-water, the NH<sub>3</sub> is first distilled in a current of air at a pressure of 4 to 5 cm of mercury. For optimum recovery of NH<sub>3</sub> the sample is adjusted to  $\approx$  pH 9.15 by the addition of a sodium metaborate-sodium hydroxide buffer soln. A diagram of the apparatus is shown. The NH<sub>3</sub> in the distillate is determined by a modification of Russell's colorimetric indophenol method (*Brit. Abstr. C*, 1945, 129). For natural fresh waters the distillation is unnecessary, but pptn. occurs when the colorimetric method is applied directly to sea-water.

W. C. JOHNSON

826. **Separation and concentration of trace metals from natural waters. Partition chromatographic technique.** D. E. Carritt (*Anal. Chem.*, 1953, 25 [12], 1927-1928).—The operation of an extraction

column, packed with a granular cellulose acetate support carrying a  $\text{CCl}_4$  soln. of dithizone, is described. The device, which was designed for the analysis of trace elements in sea water, depends on the formation of metal dithizonates and operates as a partition-chromatographic column in which the  $\text{CCl}_4$ -dithizone soln. is the immobile phase. The dithizonates of Pb, Zn, Mn, Cd, Co and Cu are all removed completely from soln. by the column. In the studies reported, the metals (except Mn, which was determined spectrophotometrically) were determined in the eluates by the polarographic method.

J. M. JACOBS

**827. Determination of electrometric properties of ground water by a field method.** R. S. Pierce (*Trans. Wisconsin Acad.*, 1953, **42**, 173-176).—The soil is excavated until slight seepage of water is evident. A galvanised-iron well point containing a platinum electrode is then inserted to a depth of 18 in. Connection is made by a KCl bridge to a calomel half-cell and readings are taken on a Beckman potentiometer.

E. G. BRICKELL

**828. Methods for determining hydrocarbons and phenols in water.** American Petroleum Institute (*Anal. Chem.*, 1953, **25** [11], 1681-1685).—Six methods for the determination of oils or phenols in water have been developed or evaluated by a group of laboratories working through the Committee on Analytical Research of the A.P.I. The i.r. method of Simard Hasegawa, Bandaruk and Headington (*Brit. Abstr. C*, 1952, **69**, and *Anal. Chem.*, 1952, **24**, 909) for oils and phenols was shown to be reliable for oil concn. as low as 0.1 p.p.m. and to have an error  $< \pm 20$  per cent. The benzene extraction method of Musante (*Brit. Abstr. C*, 1952, **151**) for oils gave high recoveries for non-volatile oil, good recoveries for oil containing kerosene and low recoveries for oil containing gasoline. The reflux-benzene extraction method of Neusbaum (private communication) for oil was considered satisfactory and has been adopted by one of the A.P.I. committees as an approved method. An extraction-pyknometer method for samples containing volatile hydrocarbons was shown to be promising (*Anal. Chem.*, in the press). A mass-spectrometer method for estimating as little as 0.1 p.p.m. of oil has not yet been thoroughly tested. A method for phenols, involving bromination of the phenols, extraction and u.v. measurement according to the method of Murray (*Anal. Chem.*, 1949, **21**, 941), has been developed, but has not yet been evaluated. The method is applicable to the determination of  $\approx 1$  pt. per  $10^6$  of phenol.

G. P. COOK

**829. Lauryl tryptose broth for coli determinations in chlorinated sewage.** C. T. Mudgett (*Sewage & Ind. Wastes*, 1953, **25** [5], 557-559).—In a study of the effectiveness of sewage chlorination, the *E. coli* count being the criterion, in addition to the determination by the standard method of planting three duplicate tubes at four dilutions in lactose broth, followed by transfer of the positive tubes to brilliant green bile for confirmation, parallel samples of chlorinated effluent were also run in lauryl tryptose. Statistical examination of the results by a graphical method shows that simple planting in lauryl tryptose broth gives results comparable to those by the standard lactose broth test with brilliant green bile confirmation.

J. M. JACOBS

**830. Determination of the gamma-isomer content of benzene hexachloride by chlorine-36 isotope dilution method.** J. T. Craig, P. F. Tryon and

W. G. Brown (*Anal. Chem.*, 1953, **25** [11], 1661-1663).—A known quantity of chemically pure radioactive  $\gamma$ -isomer of benzene hexachloride labelled with  $^{36}\text{Cl}$  is added to a sample of technical benzene hexachloride of unknown  $\gamma$ -isomer content. A sample of pure  $\gamma$ -isomer is then isolated from the mixture by extraction with perchloroethylene. The solvent is evaporated, the residue of oil is weighed, and the  $\gamma$ -isomer is crystallised from a solvent mixture of equal vol. of 1:4-dioxan and *n*-butanol. Crystallisation is repeated until crystals melting between  $112^\circ$  and  $112.8^\circ\text{C}$  are obtained. A soln. of the crystals in acetone is prepared and is counted (by a Geiger counter), the specific activity of this isolated material being compared with that of the original labelled isomer. The standard deviation of the method is  $\pm 0.2$  per cent. for  $\gamma$ -isomer content and results agree well with those by other methods. Details of the preparation of the labelled  $\gamma$ -isomer are given.

G. P. COOK

#### Agriculture and Plant Biochemistry

**831. Micro-determination of zinc in soil extracts by paper chromatography.** G. G. Lamm (*Acta Chem. Scand.*, 1953, **7** [10], 1420-1422).—Small amounts of zinc in soil extracts are separated by paper chromatography from other metals, except mercury, that form coloured complexes with dithizone by means of a mixture of *n*-butanol and 2*N* HCl (1 + 1) as solvent. The isolated zinc zone on the chromatogram is cut out and the zinc is eluted and determined colorimetrically with dithizone.  $R_F$  values for the various metals under these conditions are given.

I. JONES

**832. Field method for determination of traces of arsenic in soils. Confined spot procedure using a modified Gutzeit apparatus.** H. Almond (*Anal. Chem.*, 1953, **25** [11], 1766-1767).—A field method is described for determining as little as 5 p.p.m. of As in 0.1-g soil samples by a modified Gutzeit procedure. The wetted sample is fused with KOH which attacks the silicates and arsenic compounds without loss of volatile arsenic compounds. Zn is then added to an acidified aliquot in a modified Gutzeit apparatus (illustrated and described) and the gases are passed through lead acetate on glass wool to a confined spot on  $\text{HgCl}_2$  paper. If adequate lead acetate is used, traces of arsenic can be determined in the presence of large amounts of sulphides. Moderate amounts of antimony do not interfere. At least eight samples can be analysed in 90 min.

O. M. WHITTON

**833. Sulphosalicylic acid method for iron determination and its use in certain soil analyses.** E. Koulter-Andersson (*Kungl. Lantbr.-Högskol. Ann.*, 1953, **20**, 297-301).—Dilute a volume of a weakly acidic soln. containing 0.1 to 0.8 mg of  $\text{Fe}_2\text{O}_3$  to  $\approx 50$  ml with water, add 5 ml of 25 per cent. sulphosalicylic acid soln., then sufficient conc. aq.  $\text{NH}_3$  to change the pink-violet colour to yellow (or in the presence of interfering substances, neutralise until a yellow colour appears) and 0.5 to 1.0 ml in excess, and dilute to 100 ml. After 30 min. measure the extinction of the soln. in a photometer with a dark-blue filter or in a spectrophotometer at 430 m $\mu$ , and compare the extinction with that of a standard  $\text{Fe}^{+++}$  soln. containing 0.025  $\mu\text{g}$  of  $\text{Fe}_2\text{O}_3$  per ml prepared from a conc. soln. of  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  by oxidising the  $\text{HNO}_3$ , standardising gravimetrically, diluting and adding 5 ml of  $\text{HNO}_3$  per litre. The colour is stable for  $> 24$  hr. and is independent of

NH<sub>3</sub> concn. High concn. of Mg and Al form complexes with the reagent but do not prevent colour development. The brownish-red colour given by Mn is avoided by adding 2 to 5 ml of 5 per cent. NH<sub>4</sub>OH.HCl soln. before neutralising, and the green colour due to Cu by adding 1 to 2 drops of 5 per cent. KCN soln. Total iron in Tamm's acid oxalate and in 0.2 N H<sub>2</sub>SO<sub>4</sub> extracts of certain soils can be determined accurately and simply by this method, sometimes directly or otherwise after removal of ammonium salts and free oxalic acid.

S. C. JOLLY

See also Abstracts 797, 858.

## 5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

### General

834. **A microbalance for the determination of protein crystal densities.** F. M. Richards (*Rev. Sci. Instrum.*, 1953, 24 [11], 1029-1034).—Constructional details are given of a microbalance for the simultaneous measurement of the apparent weight and vol. of a single protein crystal immersed in its mother liquor. The balance is a single-fibre type. Crystal vol. is estimated from photomicrographs taken in two directions at right angles.

G. SKIRROW

835. **Separating funnels.** British Standards Institution (B.S. 2021 : 1953, 12 pp.).—This specification covers stoppered glass separating funnels of spherical, conical or cylindrical shape and nominal capacities of 25 to 5000 ml. Suitable dimensions are indicated for the guidance of manufacturers but they are not mandatory. If interchangeable glass stoppers are provided they must comply with the relevant provisions of B.S. 573 and the stopcocks must comply with the provisions of B.S. 1751. The tolerances on capacity for graduated separating funnels are specified.

J. M. JACOBS

836. **Weighing pipettes.** British Standards Institution (B.S. 2058 : 1953, 7 pp.).—Two types of weighing pipette for general laboratory purposes are specified, viz., Type 1, the conventional Lunge-Rey pattern, with one bulb and two stopcocks, and Type 2, a pattern with two bulbs and three stopcocks especially suitable for the analysis of aq. NH<sub>3</sub>. For those of type 1, alternative cylindrical and conical cover tubes, with interchangeable joints, are specified. The general design of each type of pipette is illustrated. The pipettes must be of good workmanship and light construction and the total wt. must be  $\geq 35$  g. The stopcocks must be carefully ground and the open ends fine-polished. The only mandatory dimension is the over-all length, which must be  $\geq 145$  mm for Type 1 and  $\geq 150$  mm for Type 2. The other dimensions are included for the guidance of manufacturers, but a weighing pipette that on visual inspection shows any gross departure from them is deemed not to comply with the specification.

J. M. JACOBS

837. **Brewers' mash flask.** British Standards Institution (B.S. 701 : 1953, 8 pp.).—The B.S. specification for "Brewers' flasks" (1936) has been revised and brought into line with other recent B.S. specifications for volumetric glassware. Dimensions (with tolerances) and requirements as to stability

of the flasks on an inclined plane are given. Calibration for contents by weighing with water filled to the 500 and 515-ml graduations may now be carried out at 27° C as well as at 20° C.

P. S. ARUP

838. **Safety switch for water-cooled systems.** W. Roth (*Anal. Chem.*, 1953, 25 [12], 1940).—A safety control for an 'unattended water-cooled mercury diffusion pump comprises a brass bellows with an outlet tube smaller in diameter than that of the inlet. The bellows is linked by a lever arm to a switch connected in series to the heater to be controlled. When water is flowing through the apparatus the bellows expand owing to the restriction of flow caused by the smaller outlet. A decrease in the rate of flow causes the bellows to contract and interrupts the heating circuit.

J. M. JACOBS

839. **Simple multi-unit magnetic stirrer.** L. A. Wollerman and R. G. Tischer (*Anal. Chem.*, 1953, 25 [12], 1942).—The power source is a small (variable-speed drive) motor with a bar magnet wired across the pulley to actuate the stirring bar. The pulley-shaft, which is a short iron rod (on which the pulley may turn freely), is inserted in a rubber stopper. The stopper is mounted in a burette clamp with a sleeve between the stopper and the pulley to allow free rotation of the pulley.

J. M. JACOBS

840. **Glass atomiser for use in chromatography.** W. J. Wingo (*Anal. Chem.*, 1953, 25 [12], 1939-1940).—The construction of a glass atomiser for spraying chromatograms with corrosive liquids is described. The device gives a spray having a small droplet size and delivers  $\approx 5$  ml of spray per min. when supplied with air at a pressure of 7 to 10 lb per sq. in.

J. M. JACOBS

841. **Magnetic pumps for corrosive gases and liquids.** F. D. Rosen (*Rev. Sci. Instrum.*, 1953, 24 [11], 1061-1062).—A sealed magnetically-operated pump suitable for circulation of corrosive gases such as UF<sub>6</sub> is described. The pump will maintain a pressure difference of 150 mm of mercury at a flow-rate of 600 ml per min.

G. SKIRROW

842. **Sampling gun for spectrographic pin samples.** H. H. Grossman, C. B. Pratt and W. S. Myers (*Anal. Chem.*, 1953, 25 [12], 1939).—A sampling gun for drawing molten Mg alloys into a borosilicate glass sampling tube is described. The tube is dipped below the surface of the molten metal and a trigger is pulled which releases a spring-loaded rod, thus causing motion of a leather-cup piston that creates the partial vacuum for the sampling.

J. M. JACOBS

843. **Water vapour saturation in gas-measuring burettes.** T. W. Stein and R. C. Reid (*Anal. Chem.*, 1953, 25 [12], 1919-1920).—In volumetric (Orsat) analysis of gas mixtures, the gas sample must have the same percentage of water vapour for each determination of a component. This is usually accomplished by keeping the gas sample saturated with water vapour. Calculations are presented to show that a clean burette, the walls of which are covered with a thin film of water, will saturate the gas very rapidly, whereas in a burette whose walls (e.g., owing to a layer of grease) are not wetted by water it is virtually impossible to saturate the gas in any reasonable length of time. Errors arising from failure to saturate the gas sample with water vapour may be large.

J. M. JACOBS

**844. Improved method of determining densities of viscous fluids with Babcock bottles.** R. S. Cass, J. B. Gregory and E. Levens (*Anal. Chem.*, 1953, **25** [11], 1773).—A method is described for the precise and rapid testing of viscous fluids at different temperatures. The pycnometers used are Babcock milk test bottles of 50-ml capacity having a wide base, a neck with 0.02-ml graduations and a vol. of 1.6 ml. The bottles can be filled with a pipette, loaded in an oven, and used for non-viscous as well as viscous fluids. Density-temperature results can be obtained over a wide range of temperatures (130° F for many fluids) without a separate weighing for each determination. Only initial (tare) and final weighings, as well as neck readings at each temperature, are necessary. Fluid densities obtained with different bottles are tabulated. They show a standard deviation of  $\pm 0.0001$  g per ml ( $\pm 0.005$  per cent.). Precision is slightly better for any particular bottle. With care, a precision of  $\pm 0.0002$  g per ml ( $\pm 0.01$  per cent.) can be attained with fluid viscosities of 1000 to 2000 centipoises at 160° to 180° F. Densities obtained by the Babcock method and by standard pycnometers agree in the third decimal place (1 part in 2000). The absolute accuracy is probably about  $\pm 0.05$  per cent. because of the additional errors involved in calibration. O. M. WHITTON

**845. A mercury-pressure safety valve.** S. H. Tucker (*Analyst*, 1954, **79**, 55).—A drawback of many types of mercury-pressure safety valves is that, owing to fall of temp. or other causes, mercury may be sucked back into the apparatus. The valve described is free from this defect, the design being such that mercury sucked back does not reach the orifice into the apparatus, but is trapped in an outer compartment where it forms an additional seal. A. O. JONES

**846. Constant-pressure device for gas flow system.** A. Charnley and G. I. Isles (*Anal. Chem.*, 1953, **25** [12], 1942).—The device described by Clow *et al.* (*J. Sci. Instrum.*, 1939, **16**, 354) is modified by incorporation of a solenoid-controlled, cone-tipped iron plunger and a seating made from a soft pencil eraser; this arrangement withstands fast vibrations for long periods. The sensitivity of the device depends on the flow rate, the backing pressure and the hole in the cut-off. The effective size of the hole can be altered by varying the height of the plunger oscillation with a rubber-tipped brass bar that can be adjusted externally by a flexible copper bellows. J. M. JACOBS

**847. Liquid manometers with high sensitivity and small time-lag.** F. A. MacMillan (*J. Sci. Instrum.*, 1954, **31** [1], 17-20).—An analysis is given of the factors responsible for time-lag in manometers owing to change of zero reading. With a null-reading inclined-tube manometer, changes of zero reading caused by variations in temp. can be eliminated by correct choice of manometer dimensions. G. SKIRROW

**848. Pneumatic gauging applied to the measurement of the bore of tube.** R. Chittleburgh, E. F. Powell and G. F. Morton (*J. Sci. Instrum.*, 1954, **31** [1], 20-22).—A stream of air is passed through the tube in which a steel ball serving as a constriction is held at any desired position by an external electromagnet. The pressure developed between the ball and a fixed constriction at the entry to the tube is a measure of the gap between the wall of the tube and the ball. G. SKIRROW

**849. Pressure chamber for micro-optical observations.** C. Turner (*J. Sci. Instrum.*, 1954, **31** [1], 9-10).—The pressure chamber (external diam. 6 cm, height 5½ cm) is designed for observing transition and melting points at pressures up to 1000 atm. and temp. up to 100° C. Pressure and temp. can be varied independently. B. J. W.

**850. An electromagnetic method of measurement of density or specific gravity of liquids.** K. R. Honick (*J. Sci. Instrum.*, 1954, **31** [1], 1-2).—A float containing a soft-iron armature is attracted either upwards or downwards through the liquid and the density is determined from a measurement of the minimum e.m.f. required to attract the float through a fixed distance. The method can be extended to remote indication or automatic recording. G. SKIRROW

**851. Simple flexibility test.** D. G. Stechert (*Anal. Chem.*, 1953, **25** [11], 1730-1733).—A simple flexibility test for rubber and plastic products is described. The test piece, originally straight, is bent into a U-shape in equipment consisting of two surfaces that are always maintained parallel to one another through a pantograph linkage. In the test a distance and force is measured, from which the flexural rigidity is easily calculated. The magnitude of the test error depends largely on the extent to which friction can be minimised in the bearings of the linkage. For the apparatus described, the error (caused by friction) is  $\approx \pm 1$  per cent. when the connecting arms and base are at an angle of 30° or less. G. P. COOK

**852. The subjective assessment of firmness. Absolute standards with steel springs.** G. W. Scott Blair (*Lab. Practice*, 1954, **3** [1], 3-9).—Experiments with steel springs to determine the factors that influence the maintenance of absolute mental standards of firmness are reported. A correct guess of the code number after selecting a spring (from a series of 8) haphazardly was found to predispose the subject to an immediate subsequent success. The schemata developed during "training" are retained for several months and even after an interval of 16 months the previous level of "judgment" was recovered in 4 to 5 days. J. M. JACOBS

**853. Machines for testing the hardness of bores and other internal surfaces.** D. Napier & Son, Ltd. Inventor: Charles L. Mitchell (Brit. Pat. 703,193, Appl. date July 27, 1951).—An indenter is mounted on a hollow support which extends into the bore to be tested. A hydraulic piston associated with the indenter is connected to a ram, located outside the bore, that applies pressure to the indenter. An optical system for viewing the indentation comprises an objective mounted on the support to lie within the bore and an eyepiece to lie outside the bore. A timing device and an indicator show when the load has been applied to the indenter for a pre-determined interval. J. M. JACOBS

## Optical

**854. Moll and solvent media for infra-red use.** J. S. Ard (*Anal. Chem.*, 1953, **25** [11], 1743-1744).—The special advantages gained by using hexachlorobutadiene as a mull system instead of paraffin oil and by using tetrachloroethylene instead of CS<sub>2</sub> as a solution system are described. G. P. COOK



**855. Problems of light-excitation in electric sparks.**

J. Van Calker (*Spectrochim. Acta*, 1953, 5 [6], 476-482).—Measurement of the variation of the intensity of the lines with time during a single spark shows that the parameters of the spark source are of fundamental importance for quant. spectrochemical analysis of an alloy. The effect of different alloy components on the line-intensity concn. curve has been studied by varying the composition of the alloys. N. E.

**856. The excitation of spectral lines of non-metals in the Pfeilsticker spark.** A. Stetter (*Spectrochim. Acta*, 1953, 5 [6], 460-468).—The decisive influence of pressure on the degree of ionisation in the Pfeilsticker-type controlled arc discharge is confirmed. An attempt is made to explain by a study of the energy levels for certain atoms why conditions in this type of discharge are suitable for the detection of non-metallic elements. The effect of changes of temp. in the discharge on the relative intensity of the lines Mn II 2576 Å and Be I 2349 Å is shown to be slight. N. E.

**857. Short period spectral intensity measurements.**

D. W. Steinhilber, H. M. Crosswhite and G. H. Dieke (*Spectrochim. Acta*, 1953, 5 [6], 436-451).—For the determination of small percentages of metals, the continuous background associated with the spark source makes it difficult to measure the intensity of the spectrum line or even to detect the line. With the d.c. arc it is possible to detect very small concn. of impurities but the arc is unstable. Details are given of an automatic switch interposed between multiplier photo-tube and recorder. The switch transmits the signal only during a short time interval, the length of which can be varied as well as the delay after an initiating signal. The ability of this device to select only the arc-like portions of the individual spark discharges makes it possible to combine the reproducibility of the controlled spark with the sensitivity of the d.c. arc. By this method improvements are effected in the determination of low concn. of Cr in steel. N. E.

**858. A constant-ratio method for spectrographic analysis by the Lundegardh flame technique.**

F. L. Ashton (*Spectrochim. Acta*, 1953, 5 [6], 426-435).—A new method called the "Constant Ratio method" is described for evaluating results; Co is used as an internal standard and a series of dilutions of the unknown is used. The method is based on the principle that greatest precision is attained when the intensity of the analysis line is the same as that of the internal standard line. This avoids the necessity for background correction. An internal-standard line is selected with an intensity similar to that of the analysis line, and an intensity-ratio curve is plotted for the unknown from which the dilution required to give an intensity ratio of one is found. Standard soln. are used to find the concn. of the element required to give that ratio and from this figure the concn. of the element in the unknown is calculated. The method is applied to the analysis of soil and plant extracts. The standard deviation for Na and K is  $\pm 1.5$  per cent. The method is compared with other procedures. N. E.

**859. The effect of carbon in the spectrographic analysis of refractory materials.**

R. Ricard (*Spectrochim. Acta*, 1953, 5 [6], 417-421).—The addition of graphite to mixtures of refractory powders volatilised in the crater of a carbon arc amplifies

the lines in the emission spectra of the less volatile elements Al, Mg, Si and Ti. A study of the spectra of mixtures of Al with graphite and of Al with  $\text{TiO}_2$  and graphite shows that  $\text{TiO}_2$  increases the intensity of the lines of Al. With sufficient graphite the lines of Al have the same relative intensity with or without  $\text{TiO}_2$ . The effect is explained by the reduction of oxides by the graphite and by the prevention by the graphite of the formation of a globule in the crater which retards the vaporisation. N. E.

**860. Improvements in [optical systems of] spectrometers.**

Perkin-Elmer Corp. (Brit. Pat. 703,530, Appl. date June 9, 1952; U.S., June 22, 1951).—The proposed absorption spectrometer comprises a monochromator provided with means for deviating part of the radiation from its normal optical path, so that the deviated and undeviated radiation falling upon the exit slit is made up of two kinds of monochromatic radiation differing in wavelength. A rotary chopping disc causes the deviated and undeviated parts of the beam to be interrupted alternately, and thus permits the use of a single detector responsive to the radiation of the two wavelengths. J. M. JACOBS

**861. An apparatus for the spectrokinetic study of rapid reactions [and] Appendix: Precision of analysis by photo-electric spectrophotometry.**

K. Dalziel (*Biochem. J.*, 1953, 55 [1], 79-90; 90-93).—A modified and more precise form of the Hartridge-Roughton rapid reaction apparatus is described in detail. The reactants are introduced at high speed through separate jets into a small chamber where rapid mixing occurs; the reacting mixture is then driven at a constant rate by a gas-pressure drive down a tube of uniform bore in which the extent of reaction or the spectral absorption of transient compounds can be measured by photo-electric spectrophotometry. Factors affecting the over-all precision of the method have been examined and are discussed. The conditions under which Beer's law may be assumed valid for extinction measurements in a cylindrical tube have been determined and the factors governing the choice of wavelength for accurate kinetic measurements are stated. The extent of a suitable reaction can be estimated to within  $\pm 1.5$  per cent. or less over the first three-quarters of its course, the limiting error arising from the instability of the relative rates of delivery of the reactants through the jets. It is shown theoretically that with turbulent flow in the observation tube the validity and precision of the method should not be affected significantly by the deviations from ideal flow. The precision of the time measurements determined by the constancy of the total flow rate is  $\pm 1$  per cent. At the highest flow rate tested (280 cm per sec. in a 2-mm bore observation tube with a driving pressure of 20 lb per sq. in.) mixing occurs to the extent of 99 per cent. in 0.005 sec. The application of the apparatus to a study of the kinetics of carbonic acid dehydration is described. A value of  $12.3 \pm 0.4$  per sec. at  $18^\circ\text{C}$  was obtained for the specific reaction rate of  $\text{H}_2\text{CO}_3 \rightarrow \text{CO}_2 + \text{H}_2\text{O}$  by spectrophotometric measurements of the rate of change of pH in acid-bicarbonate mixtures with bromophenol blue as indicator. The effect of temp. on the velocity constant in the range  $19^\circ$  to  $23^\circ\text{C}$  gave the energy of activation as 16,900 g-cal. per mol. The indicator constant for bromophenol blue was found to be 3.97 at an ionic strength of 0.05. These results support the conclusions drawn from the theoretical

considerations regarding the precision of the apparatus. Other evidence for the precision of the apparatus is presented. P. CHAPLEN

862. The Cary automatic recording spectrophotometer. A. W. S. Tarrant (*Photo-elect. Spectr. Gr. Bull.*, 1953, [6], 143-149; abstr. from *Bull. Brit. Sci. Instrum. Res. Ass.*, 1953, 8, 328).—The principle of operation and the electrical and optical parts of an automatic recording spectrophotometer are described. The accuracy of transmission and of the wavelength scales are considered. R. B. CLARKE

863. Precision recording refractometer for chromatographic analysis. N. R. Trenner, C. W. Warren and Stanley L. Jones (*Anal. Chem.*, 1953, 25 [11], 1685-1690).—An instrument that is capable of continuously plotting refractive index changes relative to any reference state in any non-opaque liquid stream against mass of the liquid is described. Linearity and zero stability in each co-ordinate are good and give a max. sensitivity of  $(3.54 \pm 0.04) \times 10^{-6}$  refractive index units per division for the refractive index co-ordinate and  $56 \pm 5$  mg per division for the mass co-ordinate. With organic compounds in acetone soln., the refractive index sensitivity corresponds to a change in concn. of  $\approx 0.02$  mg per ml of acetone per scale division. G. P. COOK

864. A specimen screening aperture for the electron microscope. R. S. Page and A. W. Agar (*J. Sci. Instrum.*, 1954, 31 [1], 27).—The aperture—a 0.002 in. hole in a copper disc  $\frac{1}{4}$  in. in diam.—is attached to an L-shaped piece of brass on the end of a rod-type carrier, which controls the position of the aperture. Energy transferred to the specimen can be reduced and confined to the observed area, scattered electrons from areas outside of the field of view cannot enter the objective lens and the contamination rate of the objective aperture is reduced. B. J. W.

865. A developing tank for the uniform development of spectrographic film. F. Woutman (*Spectrochim. Acta*, 1953, 5 [6], 469-470).—Existing developing machines or tanks (i) do not always give uniformity of development because of systematic differences in the flow of developer or (ii) require a large vol. of developer. An apparatus with a tank capacity of 250 ml is described which gives reproducible and uniform development of 35-mm film in strips up to 75 cm long. N. E.

### Thermal

866. A new method of improving furnace temperature control. W. James (*J. Sci. Instrum.*, 1954, 31 [1], 23-25).—The modified bridge circuit described has an indirectly heated thermistor that can be used either to reduce the temp. differential of a resistance-thermometer on-off type temp. controller, or to convert such a controller for time-cycle proportional operation. G. SKIRROW

867. Vapour pressure ebulliometer for millilitre samples. S. R. Hoover, H. John and E. J. Mellon (*Anal. Chem.*, 1953, 25 [12], 1940-1941).—A semi-micro ebulliometer described constitutes a simple adaptation of the Cottrell principle. It was devised for the study of the v.p.-temp. relations of N-acetyl amino-acids where measurements at 75° to 250°C and pressures of 2 to 100 mm had to be made on samples smaller than 2 ml. J. M. JACOBS

868. Thermogravimetric precision analysis. P. Spinetti (*Ric. Sci.*, 1953, 23 [11], 2009-2014).—Apparatus for thermogravimetric analysis is described which permits a direct recording of wt. variation of a sample subjected to a strictly linear temp. rise and kept in a controlled atm. The main sources of errors are discussed and some recordings obtained with the apparatus are reproduced. S. K. LACHOWICZ

### Electrical

869. Hydrogen fluoride solvent system. Apparatus for polarographic studies. Rotating electrode. J. W. Sargent, A. L. Clifford and W. R. Lemmon (*Anal. Chem.*, 1953, 25 [11], 1727-1729).—The construction and design of apparatus that can handle HF solutions conveniently without corrosion or contamination is described. The equipment is made from Kel-F (polytrifluorochloroethylene), which permits visual observations and allows solutions acidic towards HF to be studied. Special reference is made to its applicability in polarography and the apparatus has been used to study anodic reactions in liquid HF by use of a micro-electrode of Ni rotating at 800 r.p.m. The current-voltage curves obtained are time dependent but are reproducible if the same time interval is used. G. P. COOK

870. Reference electrode for potentiometric titrations in glacial acetic acid. R. A. Glenn (*Anal. Chem.*, 1953, 25 [12], 1916-1917).—When the Ag-AgCl electrode (cf. Fritz, *Brit. Abstr. C*, 1951, 49) is used in conjunction with this glass electrode in a continuous automatic titration apparatus, a reversal in the change of the observed e.m.f. occurs in the region of the end-point. This reversal in the e.m.f. curve is eliminated by isolating the Ag-AgCl electrode in a separate compartment that makes electrical contact with the solution being titrated by means of a liquid-liquid junction through a ground-glass sleeve. J. M. JACOBS

871. Current supply at constant intensity for the [coulometric] titration of hydrochloric acid solutions. J. Badoz-Lambling (*Anal. Chim. Acta*, 1953, 9 [5], 455-461).—A detailed description is given of a device for the supply of current at constant intensity for coulometry. By the use of this device and the electrolytic cell also described, it is possible to titrate 0.05 N HCl with an accuracy of 0.2 per cent. W. C. JOHNSON

872. Long-life Geiger counters. H. Sherman and J. R. Heitzler (*J. Sci. Ind. Res., A, India*, 1953, 12 [8], 370-372).—A summary is given of the characteristics of self-quenching Geiger counters. A Geiger counter of extremely long life can be made by using chlorine and bromine as the filling gases. Certain disadvantages are associated with the halogen-filled tube for some kinds of work. Organic vapour-filled counters have only a limited life. Ammonia-filled counters have a shorter life than halogen-filled ones and lack the good characteristics of the organic vapour-filled counter. H. T. J. CHILTON

873. A simple mass spectrometer for analysis of stable tracer elements. E. H. Cooke-Yarborough and M. C. B. Russell (*J. Sci. Instrum.*, 1953, 30 [12], 474-480).—A 180° mass spectrometer is described suitable for the determination of isotope abundance. Constructional details and circuit diagrams are given. G. SKIRROW

## ANALYTICAL ABSTRACTS

### Translations

The following papers of interest to analytical chemists have been translated into English. Copies of these papers can be obtained from Consultants Bureau, 152, West 42nd Street, New York 18, N.Y., U.S.A. Each translation costs \$7.50 and orders should state title, author(s) and English page number. The English page number is given in parentheses after the Russian page number.

These translations can also be seen in the library of the Chemical Society, Burlington House, London, W.1.

#### J. Anal. Chem., U.S.S.R.—

The quantitative determination of halogen-organic compounds of the aliphatic series—A. K. Ruzhentseva and V. V. Kolpakova, 1952, 7 [2], 71 (81); *Brit. Abstr. C*, 1952, 488.

The quantitative colorimetric determination of epihydrin aldehyde formed during the oxidation of fats—N. S. Drozdov and N. I. Materanskaya, 1952, 7 [2], 74 (85); *Brit. Abstr. C*, 1952, 492.

The quantitative determination of the dehydration of certain alcohols—N. Z. Kotelkov and K. P. Kotelkova, 1952, 7 [2], 78 (89); *Brit. Abstr. C*, 1952, 485.

Specific reactions and methods in organic analysis. 1. The detection and quantitative determination of traces of piperidine in the presence of pyridine—L. M. Kulberg and I. S. Mustafin, 1952, 7 [2], 84 (97); *Brit. Abstr. C*, 1952, 490.

A colour reaction of dichloro-ethane—V. I. Kuznetsov and Z. M. Pimenova, 1952, 7 [2], 89 (103); *Brit. Abstr. C*, 1952, 488.

Reactions for the identification of dichloro-ethane—V. A. Nazarenko and N. V. Lapkina, 1952, 7 [2], 92 (107); *Brit. Abstr. C*, 1952, 487.

Rapid methods of micro-elementary analysis. V. A rapid method for the determination of carbon and hydrogen—M. O. Korshun, 1952, 7 [2], 96 (111); *Brit. Abstr. C*, 1952, 520.

Rapid methods of micro-elementary analysis. VI. The mechanism of the formation of silver sulphate during the simultaneous determination of carbon, hydrogen and sulphur in organic compounds—M. O. Korshun, 1952, 7 [2], 101 (117); *Brit. Abstr. C*, 1952, 520.

Rapid methods of micro-elementary analysis. VII. A new method for the simultaneous determination of carbon, hydrogen and sulphur in organic compounds containing C, H, O, N and S—M. O. Korshun and N. S. Sheveleva, 1952, 7 [2], 104 (121); *Brit. Abstr. C*, 1952, 520.

A new method for the quantitative determination of nitrogen in organic compounds—P. N. Fedoseev and N. P. Ivashova, 1952, 7 [2], 112 (129); *Brit. Abstr. C*, 1952, 520.

A new method for the quantitative determination of sulphur, and for the simultaneous determination of sulphur and nitrogen in organic compounds—P. N. Fedoseev and N. P. Ivashova, 1952, 7 [2], 116 (133); *Brit. Abstr. C*, 1952, 520.

Salicylideneamines in analysis. 4. Tricyclic inner complex salts of copper and nickel—A. P. Terentyev, E. G. Rukhadze and Z. A. Fadeeva, 1952, 7 [2], 120 (137); *Brit. Abstr. C*, 1952, 474.

A new group of reagents for boric acid—I. M. Korenman and F. R. Sheyanova, 1952, 7 [2], 128 (145); *Brit. Abstr. C*, 1952, 477.

A comparison of the characteristics of certain ion-exchange resins—D. I. Ryabchikov, M. M. Senyavin and K. V. Filippova, 1952, 7 [3], 135 (153); *Brit. Abstr. C*, 1953, 44.

Amperometric titration of nickel with dioximes—V. M. Peshkova and Z. A. Gallai, 1952, 7 [3], 162 (171); *Brit. Abstr. C*, 1953, 8.

The conductimetric titration of zinc with barium hydroxide—V. V. Udovenko and G. B. Pasovskaya, 1952, 7 [3], 158 (177); *Brit. Abstr. C*, 1953, 107.

The conductimetric determination of potassium by means of sodium picrate—V. V. Udovenko and G. B. Pasovskaya, 1952, 7 [3], 161 (181); *Brit. Abstr. C*, 1953, 1.

The chronometric determination of electrolyte concentrations. 1. Determination of the concentrations of thiosulphate and acid in their reaction mixtures—E. N. Ponomareva, 1952, 7 [3], 163 (183); *Brit. Abstr. C*, 1953, 5.

The chronometric determination of electrolyte concentrations. 2. Determination of sulphite and iodate concentrations in their acidified reaction mixtures—E. N. Ponomareva, 1952, 7 [3], 168 (189); *Brit. Abstr. C*, 1953, 5.

The intermediate potential jump during the potentiometric titration of solutions of aluminium salts with sodium fluoride—I. L. Teodorovich, 1952, 7 [3], 175 (197); *Brit. Abstr. C*, 1953, 4.

## ANALYTICAL ABSTRACTS

A demountable X-ray tube with a reverse ray path for X-ray spectral analysis—I. D. Shevaleevsky 1952, 7 [3], 183 (207); *Brit. Abstr. C*, 1953, 227.

New apparatus for electrochemical methods of analysis—V. A. Zarinsky, 1952, 7 [3], 185 (211); *Brit. Abstr. C*, 1953, 44.

The use of the ultra-violet in analytical chemistry. 2. Qualitative microchemical analysis in the ultra-violet—K. P. Stolyarov, 1952, 7 [4], 195 (221); *Brit. Abstr. C*, 1953, 141.

Use of the liquid interferometer in quantitative analysis. III. The study of dilute solutions of hydrolysable salts—I. L. Kukhtevich and A. M. Zanko, 1952, 7 [4], 201 (229); *Brit. Abstr. C*, 1953, 140.

Thermodynamic criteria of the applicability of chemical reactions in volumetric analysis—K. B. Yatsimirsky, 1952, 7 [4], 206 (235); *Brit. Abstr. C*, 1953, 140.

A colorimetric method for the determination of iron—M. Ya. Shapiro, 1952, 7 [4], 214 (243); *Brit. Abstr. C*, 1953, 114.

The use of diantipyrylphenylmethane in analytical chemistry. 2—S. I. Gusev and R. G. Beiles, 1952, 7 [4], 219 (249); *Brit. Abstr. C*, 1953, 113.

A colour reaction for the rare-earth elements—V. I. Kuznetsov, 1952, 7 [4], 226 (225); *Brit. Abstr. C*, 1953, 108.

Detection of certain rare-earth elements when present together—L. M. Kulberg and M. N. Ambrozhy, 1952, 7 [4], 233 (263); *Brit. Abstr. C*, 1953, 108.

The reaction between the bismuth ion and benzidine—G. I. Barannikov, 1952, 7 [4], 239 (269); *Brit. Abstr. C*, 1953, 110.

Determination of minimal quantities of arsenic in organic compounds by means of divalent chromium solution—P. P. Shatko, 1952, 7 [4], 242 (273); *Brit. Abstr. C*, 1953, 127.

A method for the determination of small amounts of copper—E. M. Skibina, 1952, 7 [4], 244 (275); *Brit. Abstr. C*, 1953, 105.

A cryoscopic method for the determination of arenes (aromatic hydrocarbons) in ligroin, kerosene and gas oil petroleum fractions of straight distillation—M. D. Tilicheev and N. A. Okanshivich, 1952, 7 [5], 259 (293); *Brit. Abstr. C*, 1953, 160.

A general method for the investigation of co-precipitation and absorption by means of labelled atoms—V. B. Miller, M. B. Neuman and L. A. Sazonov, 1952, 7 [5], 269 (303); *Brit. Abstr. C*, 1953, 196.

A colour reaction for fluoride ions, using titanium-chromotropic acid reagent—A. K. Babko and P. V. Khodulina, 1952, 7 [5], 281 (317); *Brit. Abstr. C*, 1953, 155.

Quantitative analysis of glasses of the system  $\text{Na}_2\text{O}-\text{CaO}-\text{SiO}_2$  on the basis of their physico-chemical properties—T. A. Popova, 1952, 7 [5], 285 (321); *Brit. Abstr. C*, 1953, 159.

The quantitative determination of tervalent iron by means of tetraborate—M. B. Shchigol and N. B. Burchinskaya, 1952, 7 [5], 289 (325); *Brit. Abstr. C*, 1953, 157.

New volumetric and gravimetric methods for the determination of cadmium—V. I. Kumov, 1952, 7 [5], 301 (337); *Brit. Abstr. C*, 1953, 147.

Determination of cuprous oxide, cupric oxide and metallic copper in copper powders—V. N. Podchainova, 1952, 7 [5], 305 (343); *Brit. Abstr. C*, 1953, 146.

The use of morpholine in analytical chemistry—M. V. Rodina, 1952, 7 [5], 312 (349); *Brit. Abstr. C*, 1953, 153.

Determination of small amounts of nitrogen in gases—A. E. Romanushkina, S. L. Kiperman and M. I. Temkin, 1952, 7 [5], 314 (351); *Brit. Abstr. C*, 1953, 151.

An apparatus for the determination of nitrogen in gaseous mixtures—I. G. Lesokhin, B. A. Kopylev and A. Ya. Averbukh, 1952, 7 [5], 316 (353); *Brit. Abstr. C*, 1953, 151.

ERRATUM.—March (1954) issue, abstract 573; line 7.  
For saponifiable read unsaponifiable.



## ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

|                            |                      |                                   |                   |
|----------------------------|----------------------|-----------------------------------|-------------------|
| alternating current        | a.c.                 | micro-litre                       | $\mu$ l           |
| ampere                     | amp.                 | micron                            | $\mu$             |
| Angstrom unit              | $\text{\AA}$         | milliampere                       | mA                |
| anhydrous                  | anhyd.               | milligram                         | mg                |
| approximate, -ly           | approx.              | millilitre                        | ml                |
| aqueous                    | aq.                  | millimetre                        | mm                |
| atmosphere, -ic            | atm.                 | millivolt                         | mV                |
| atomic                     | at.                  | minimum                           | min.              |
| boiling-point              | b.p.                 | minute (time)                     | min.              |
| British thermal unit       | B.Th.U.              | molar (concentration)             | M                 |
| calculated                 | (calc.)              | molecule, -e, -ar                 | mol.              |
| calorie (large)            | kg-cal.              | normal (concentration)            | N                 |
| calorie (small)            | g-cal.               | number                            | no.               |
| centimetre                 | cm                   | observed                          | (obs.)            |
| coefficient                | coeff.               | organic                           | org.              |
| concentrated               | conc.                | ounce                             | oz.               |
| concentration              | concn.               | part                              | pt.               |
| constant                   | const.               | patent                            | pat.              |
| corrected                  | (corr.)              | parts per million                 | p.p.m.            |
| critical                   | crit.                | per cent. wt. in wt.              | per cent. w/w     |
| crystalline                | } cryst.             | per cent. wt. in vol.             | per cent. w/v     |
| crystallised               |                      | per cent. vol. in vol.            | per cent. v/v     |
| cubic                      | cu.                  | potential difference              | p.d.              |
| current density            | c.d.                 | pound                             | lb                |
| cycles per second          | c.p.s.               | precipitate                       | ppt.              |
| decompos- ing, -ition      | (decomp.)            | precipitated                      | pptd.             |
| density                    | $\rho$               | precipitating                     | pptg.             |
| density, relative          | $\rho$ or wt. per ml | precipitation                     | pptn.             |
| derivative                 | deriv.               | preparation                       | prep.             |
| dilute                     | dil.                 | qualitative, -ly                  | qual.             |
| direct current             | d.c.                 | quantitative, -ly                 | quant.            |
| distilled                  | dist.                | recrystallised                    | recryst.          |
| electromotive force        | e.m.f.               | refractive index                  | $n_D^{20}$        |
| electron-volt              | eV                   | relative humidity                 | R.H.              |
| equivalent                 | equiv.               | revolutions per minute            | r.p.m.            |
| experiment, -al            | expt.                | saponification value              | sap. val.         |
| gram                       | g                    | saturated calomel electrode       | S.C.E.            |
| gram-molecule              | mole                 | second (time)                     | sec.              |
| half-wave potential        | $E_1$                | soluble                           | sol.              |
| horse-power                | h.p.                 | solution                          | soln.             |
| hour                       | hr.                  | specific gravity                  | sp. gr.           |
| hydrogen ion concentration | $[\text{H}^+]$       | specific rotation                 | $[\alpha]_D^{20}$ |
| hydrogen ion exponent      | pH                   | square centimetre                 | sq. cm            |
| inch                       | in.                  | standard temperature and pressure | s.t.p.            |
| indefinite                 | indef.               | temperature                       | temp.             |
| infra-red                  | i.r.                 | ultra-violet                      | u.v.              |
| insoluble                  | insol.               | vapour density                    | v.d.              |
| kilogram                   | kg                   | vapour pressure                   | v.p.              |
| kilovolt                   | kV                   | volt                              | V                 |
| kilowatt                   | kW                   | volume                            | vol.              |
| liquid                     | liq.                 | watt                              | W                 |
| maxim -um, -a              | max.                 | wavelength                        | $\lambda$         |
| melting-point              | m.p.                 | weight                            | wt.               |
| microgram                  | $\mu$ g              |                                   |                   |

In addition the following symbols are used—

|                    |             |                                |             |
|--------------------|-------------|--------------------------------|-------------|
| greater than       | >           | less than                      | <           |
| not greater than   | $\nlessgtr$ | not less than                  | $\nlessgtr$ |
| is proportional to | $\propto$   | of the order of, approximately | $\approx$   |

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicals are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, e.g.,  $\text{Cu}^{++}$ ,  $\text{Al}^{+++}$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{--}$ . Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, e.g., ferric iron becomes  $\text{Fe}^{\text{III}}$  and cuprous copper  $\text{Cu}^{\text{I}}$ .

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